THE EVALUATION OF USING MICRONIZED BARITE AS A COMPLETION FLUID WEIGHTING MATERIAL IN HPHT WELLS

BY

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This work is dedicated to my strength, lovely family.

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HPHT: High Pressure High Temperature

DTPA: Di-ethylene Tri-amine Penta acetic Acid

XRD: X-Ray Diffraction

SEM: Scanning Electron Microscope

XRF: X-Ray Fluorescence

ABSTRACT

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Thesis Title : The Evaluation of Using Micronized Barite as a Completion Fluid Weighting Material in HPHT Wells

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Completion and workover fluids are one of the prominent factors that play a great role in the success of completion and workover operations. Since these fluids have a great impact on personnel safety, environment, wells productivity, and the total cost of the operation, great efforts should be put into selecting the optimal fluid to accomplish the required job.

Completing high pressure high temperature wells is one of the challenging jobs that require formulating a special type of fluids due to the complexity and the critical downhole conditions of high pressure high temperature wells. This complexity and the new technical developments on drilling and completion operations pushed the industry towards the development of new formulations and implementation of advanced technologies to meet all the requirements and the use of conventional drill-in fluids, barite-weighted fluids, was discontinued. However, some of the proposed fluids have environmental issues while others are costly.

Since conventional drill-in fluids have high density, are environmentally friendly and relatively inexpensive, this work aims to evaluate micronized barite as a weighting material to resolve the problems associated with conventional drill-in fluids, i.e. formation damage

and fluid stability, in order to use these fluids as completion and workover fluids in HPHT wells.

In addition to the commercial sample of barite, different sizes of barite, i.e. 106-75 µm, 75-40 µm, and less than 40µm, were prepared using sieve analysis. Moreover, a sample of micronized barite was prepared by reducing barite particle size to a few microns using ball milling machine after optimizing the operating parameters of ball milling machine.

Afterwards, solubility tests were conducted to study the effect of particle size on barite removal using a recently developed formulation, DTPA with potassium carbonate as a catalyst. The effect of barite particle size on the rheological properties of the drill-in fluids was investigated at 120 and 250°F. Barite particle size effect on fluids stability was also investigated using zeta potential measurements and static sag test.

Furthermore, a new formulation using different proportions of bridging agent (calcium carbonate) was designed to mitigate the resulted formation damage by minimizing fluid filtrate and solid particles invasion into the formation. The new formulation was evaluated to ensure its capability to properly accomplish the required job.

As a result of barite particle size reduction, solubility test results showed a good enhancement in barite removal due to the increase of chemical reaction surface area, and HPHT filtration test confirmed these results and showed an improvement of around 5% in filter cake removal efficiency.

Moreover, reducing barite particle size showed a thickening behavior in rheology of completion fluids and as the temperature increases, the thickening behavior becomes more noticeable.

Finally, zeta potential measurements showed a good enhancement in stability with micronized barite for a pH range greater than 8, while the other sizes of barite were lying in the unstable range, i.e. -30 to +30 mv. While, in contrast, sag test showed insignificant enhancement in fluid stability as barite particle size was reduced to micronized size with a sag factor of 0.546 as compared to 0.563 for the normal size at 230°F.

ملخص الرسالة

الاسم الكامل: عبدالمجيد كمال عبدالرحمن محمد

عنوان الرسالة: تقييم ودراسة البارايت ذو الجزيئات الصغيرة كمادة لزيادة كثافة السوائل لإستكمال الآبار ذات الضغط و درجات الحرارة المرتفعة

التخصص: هندسة النفط

تاريخ الدرجة العلمية: يناير 2016

سوائل إستكمال وصيانة الآبار هي واحدة من العوامل البارزة التي تلعب دورا كبيرا في نجاح عمليات إستكمال وصيانة الآبار. وبما أن هذه السوائل يكون لها تأثير كبير على سلامة الأفراد والبيئة والآبار الإنتاجية، والتكلفة الإجمالية للعملية، يجب وضع جهودا كبيرة في اختيار السائل الأمثل لإنجاز العمل المطلوب.

إستكمال الآبار ذات الضغط و درجة الحرارة المرتفعة هي واحدة من الوظائف الصعبة التي تتطلب تقديم نوع خاص من السوائل نظرا للظروف الحرجة في اسفل البئر، ارتفاع ضغط المكامن و ارتفاع درجة الحرارة. هذا التعقيد والتطورات التقنية الجديدة في عمليات الحفر والاستكمال دفعت الصناعة نحو تطوير السوائل وتنفيذ تقنيات متقدمة لتلبية جميع المتطلبات الجديدة واستخدام سوائل الحفر التقليدية، البارايت، قد توقف. ومع ذلك، فإن بعض السوائل المقترحة لديها مشاكل بيئية في حين أن الأخرى مكلفة.

السوائل التقليدية لديها كثافة عالية، وصديقة للبيئة وغير مكلفة نسبيا، ويهدف هذا العمل إلى تقييم البارايت ذو الجزيئات الصغيرة كمادة مضافة لزيادة كثافة السوائل لحسم المشاكل المرتبطة بسوائل الحفر التقليدية، أي ضرر خفض الانتاجية واستقرارية هذه السوائل، وذلك للاستفادة من هذه السوائل في إنجاز وصيانة الآبار ذات الضغط و درجة الحرارة العالية.

تم تجهيز عينات من البارايت بجزيئات مختلفة الحجم و ذلك عن طريق الغربال و الطحن و ذلك لدراسة تأثير حجم الجزيئات في استقرارية السوائل و في إزالة هذه السوائل لرفع الانتاجية. بعد ذلك، تم إجراء اختبار الذوبانية لدراسة تأثير حجم الجزيئات على إزالة البارايت باستخدام صيغة وضعت مؤخرا، باستخدام السوائل المخلبية (DTPA) مع كربونات البوتاسيوم كعامل محفز. كما تمت دراسة تأثير تقليل حجم الجزيئات على خواص الجريان (Rheology).

وعلاوة على ذلك، تم تصميم صياغة جديدة باستخدام البارايت ذو الجزيئات الصغيرة مع الإضافات المناسبة وذلك لتخفيف الضرر الناتج عن غزو الجسيمات الصغيرة للطبقة المنتجة. تم تقييم صياغة جديدة في ظل ظروف الضغط و الحرارة العالية لضمان قدرتها على إنجاز المهمة المطلوبة بشكل صحيح.

و من نتائج هذه الدراسة أن تقليل حجم الجزيئات أسهم في رفع كفاءة إزالة البارايت و بالتالي رفع انتاجية الابار كما كان عاملاً فعّالاً في تحسين استقرارية هذه السوائل.

CHAPTER I

INTRODUCTION

1.1 Background

Completion and workover fluids can be defined as the fluids that are mainly used to maintain and control the well pressure during completion and workover jobs, such as perforation, sand consolidation operations, gravel packing, or any other completion techniques. Several types of completion fluids can be introduced into the well, however, this fluid should be dense enough to balance the formation pressure, the higher formation pressure we encounter the denser fluid is required.

Moreover, completion and workover fluids must also be capable of suspending the free solids that are required in some operations, such as under reaming and gravel packing, additionally, in well killing and cementing, for instance, it must be able to replace other fluids in the wellbore.(Patton and Phelan, 1985) Efficiency, safety, cost, and the impact on well productivity are the main factors should be put into consideration while selecting and designing any completion fluid.

Different fluids were used to maintain that positive differential pressure such as, salt water, sodium chloride, calcium chloride, and zinc chloride. Nevertheless, each fluid has some drawbacks which require formulating special types of completion fluids for any reservoir conditions. For instance, although salt water is economical to be used as a

completion fluid but low densities can be obtained, only up to 10 lb/gal, which cannot be used safely in deep reservoirs due to the high formation pressures.(Paul et al., 1974)

Calcium chloride can also be used with a density up to 11.6 lb/gal and some insoluble weighting materials are used, to accomplish higher densities but, in facts, those materials cause some problems such like permeability reduction which, in some severe cases, leads to losing the production.(Paul et al., 1974) **Table 1.1** shows the common used brine as completion fluids and their densities. In fact, some fluids are damaging, corrosive, very costly or even environmentally degrading. Hence, one question that arises, what is the optimum completion fluid to be used in HPHT wells.

Table 1.1: The Common Used Brine as Completion Fluids (Caenn et al., 2011)

Fluid	Final Density, ppg
Sodium Chloride	8.4 to 10
Calcium Chloride	8.4 to 11.63
Sodium Chloride/ Calcium Chloride	9 to 11.23
Calcium Chloride/ Calcium Bromide	10.83 to 13.33
Calcium Bromide/ Zinc Bromide	13.33 to 18.33

1.2 High Pressure High Temperature (HPHT) Wells

Any oil or gas well has a pore pressure gradient and temperature greater than 0.8 psi/ft and 300 ^{0}F, respectively, is classified as high pressure high temperature well(Schlumberger, n.d.; Smithson, 2016). Completing high pressure high temperature wells requires formulating a special fluid which has a good stability under downhole conditions and a high density to control the wells with less solids that could cause serious problems such as, permeability reduction, solids settling, and in some cases when gel fluids are used to remain the solids suspended; a loss in viscosity control might result

especially with uncertain bottom-hole temperatures. Generally, two choices of completion fluids were reported in the literature, either utilizing the conventional drilling fluids or using heavy brine formulations.

1.3 Formation Damage

Formation damage is defined as any interaction between drilling, completion, or stimulation fluids and the formation/formation fluid, that might cause an impairment to the reservoir, i.e. permeability reduction in the near-wellbore area. Many fields and research studies have assured that selecting the inappropriate completion fluid or additives is one of the main causes of formation damage that leads to permeability reduction in the vicinity of the wellbore and in some severe cases it might lead to losing the production. Basically, formation damage due to completion fluids occurs from the filtrate invasion into the formation, thus the only way to significantly mitigate the damage is to reduce the filtrate to the least possible amount using the suitable additives and formulation. (Reed, 1989) Formation damage occurs in the forms of:

- Clay swelling
- Fines migration
- Emulsion and water blocks
- Scales precipitation
- Relative permeability effect
- Dissolution

- Plugging the formations due to improper operation conditions or introducing plugging materials. (Eaton and Smithey, 1971; Millhone, 1983)

Generally, there are three techniques to avoid formation damage: underbalance, where there is no fluid invasion into the formation, balanced, where a little fluid invasion is expected, and overbalance with appropriate selection of non-damaging, degradable fluids.(Millhone, 1983)

1.4 Micronized Barite, BaSO4

Micronized barite could be defined as barite that has an average particle size of a few micrometers. Basically, barite is one of the most common used weighting materials for conventional drilling fluids. It has been used since 1920s due to its high density, 4.5 g/cm^3, which makes it a good candidate to control high pressure high temperature wells. Moreover, it is easy to handle and relatively not costly. Nevertheless, its use in completion fluids was stopped due to the formation damage and stability issues. (Patton and Phelan, 1985) **Table 1.2** shows the most common used weighting materials and their densities.

Table 1.2: Common Weighting Materials Densities (Caenn et al., 2011)

Weighting Material	Density, g/cm^3
Calcite	2.7
Aragonite	2.8
Iron carbonate	3.9
Zinc carbonate	4.4
Barite	4.5
Ilmenite	4.5
Manganese tetroxide	4.7
Iron oxide	5.1
Zinc oxide	5.6
Zirconium oxide	5.7
Lead carbonate	6.6

CHAPTER II

LITERATURE REVIEW

Completion and workover fluids play a great role in the success of the completion and workover fluids. Since, producing formations, rig personnel, and the environment are exposed to these fluids thus, great efforts should be put while selecting and designing completion and workover fluids.

In general, completion and workover fluids can be classified into two main categories: clear fluids (solid-free fluid) and solid-weighted fluids. The difference between these categories is the presence of solid particles as weighting material to increase the fluid density. Fluid efficiency, compatibility with the formation, environmental impact, and the cost are the main factors should be considered before selecting and designing completion and workover fluids to ensure their ability to successfully accomplish the required job.

Fluid efficiency is the capability of the fluid to control the well throughout completion and workover operations, fluid stability under downhole conditions (i.e. pressure and temperature, and well geometry and inclination), and the ability of the fluid to displace the other fluid in the wellbore (rheology).

2.1 Conventional Drilling Fluids

Since fluid density can reach 20 ppg, conventional drilling fluids were used as completion and workover fluids. Three systems were used, water-based, oil-based, and emulsion mud. Consequently, formation damage resulted in many cases.

In 1985, Patton and Phelan reported that the main problem of using conventional drilling fluids as completion fluids is the high solid content and these solid particulates were found to be the main cause of formation damage in so many cases in the literature, and therefore, their use was stopped. (Patton and Phelan, 1985) Another problem of using the drilling fluids is the possibility of solid particles settlement, sag effect, which would not solely lead to losing their efficiency to control the well, but these solid particles will also interfere with completion and workover operations. (Scott et al., 2004)

Barite sag, or solids sag, is a complex phenomenon and very likely to occur in deviated wells especially in low-shear cases(Hanson et al., 1990). Although it is very difficult to simulate, but a good optimization and monitoring for mud rheological properties, sound strategies, and training for drilling personnel would help manage solids sag and mitigate its consequences. (Scott et al., 2004)

Many cases of the sag effect were documented in the literature, for instance, during completing the first well in Hulder field in the North Sea using barite- weighted oil-based mud, a severe kick was encountered. It occurred because of barite sag, and well geometry contributed in the sag effect. (Saasen et al., 2002)

Alabdullatif et al. introduced a new formulation of water base mud to kill the over-pressured formations. They combined Mn_3O_4 to the normal weighting material, barite, to overcome barite sag tendency that was encountered in previous operations. Mn_3O_4 was very effective in enhancing sag performance and minimizing the risk of losing well control especially after a long time of static condition. (Alabdullatif et al., 2015)

In summary, if these two problems, formation damage and sag effect, were solved, the conventional drilling fluids would be one of the best choices of well servicing fluids due to their relatively low cost and the possibility of having high density fluid that could perfectly control high pressure wells.

2.2 Heavy Brines

In general, heavy brine could be classified into two categories, particulate-weighted fluids which are formulated by adding acid-soluble weighting agents and/or without non-damaging polymers, and clear fluids (solids-free fluids). (Paul et al., 1974)

In 1973, Paul J. R. and Plonka J. H. proposed "calcium bromide/ calcium chloride" as a solution to high pressure wells. They reported that the proposed solution of calcium bromide/ calcium chloride could be prepared with a range of density from 11.6 lb/gal and up to 15.1 lb/gal with no solids (**Table 2.1**). Additionally, it has been proved that calcium bromide is corrosion resistive and environmentally friendly. Although the cost of calcium bromide/ calcium chloride is less than double of the cost of conventional drilling muds but, for instance, one dead well or a fishing job for a stuck packer in an offshore well that might result from using that conventional drilling muds would be too much expensive than using calcium bromide/ calcium chloride as a completion fluid. (Paul and Plonka, 1973)

In 1979, from the case of Cal Canal field, Conners and Bruton confirmed that calcium bromide/calcium chloride solution can be successfully used as a drill-in fluid. Ordinary drilling mud was replaced by calcium bromide/calcium chloride to drill the producing

formation in around 18 wells and almost no formation damage was recorded. They also concluded that the high cost of using calcium bromide/calcium chloride could be minimized by experience and correct practices, especially with a large number of wells. Drilling with calcium bromide/calcium chloride requires effective solid removal equipment and prior thorough study to determine its applicability. (Conners and Bruton, 1979)

Table 2.1: Solids Free Completion Fluids- Case Histories (Paul and Plonka, 1973)

Well	A	B	C	D	E to H
Location	Offshore Louisiana	Offshore Louisiana	Offshore Louisiana	Offshore Louisiana	Offshore Louisiana
Depth, ft	8,000	8,500	10,000	10,500	8,000 Range
Well Status	New Workover	New	New	New	Workover
Completion Fluid:					
Type	CaBr2/CaCl2	CaCl2	CaBr2/CaCl2	CaBr2/CaCl2	CaBr2/CaCl2
Density, ppg	15	11.7	12.5	12.5	12
Volume, bbls	35	1000	125	150	100
Oil or Gas	Oil	Oil	Oil	Oil	Oil
Reservoir Pressure, psi	5,800	5,100	7,000	7,200	4,400
Casing Size	7"	7 5/8"	-	-	7 5/8 - 9 5/8"
Tubing Size	2 3/8"	2 3/8" Dual	3 1/2"	3 1/2"	2 3/8 - 2 7/8''
Packer	Yes	Yes	No	No	Yes
Purpose	To control pressure during gravel pack procedure; no injection of solids to formation	for packer hold down fluid during perforating and completing of well; gravel pack	To control reservoir pressure during plastic sand consolidation and well completion		To control pressure during sand clearout and general workover operations
Results	Successful	Successful	Successful	Successful	Successful

Another case of using calcium bromide/calcium chloride brine as a completion fluid was found in the literature. It has been used in the Gulf Mexico by Amoco Production Company and two problems were encountered: 1) High fluid losses at the surface and into the formation that led to an extreme increase in the cost of the completion job (**Figure 2.1**), 2) losing some weight of the completion fluid due to hydration, mixing with air and formation fluids, and crystallization due to the bottom hole temperature and that would result in losing well control. Thus, Zinc bromide was added to the solution to increase the fluid density with 200 psi overbalance in order to maintain the well control

during the whole period of completion operation. Nevertheless, zinc bromide was found to be environmentally hazardous and causes skin diseases and loss of vision when eyes are exposed to it, thus all personnel on the floor were required to use safety goggles. (Spies et al., 1983)

To reduce the fluid losses into the formation, various types of polymers have been used to maintain the desirable viscosity. Darlington et al. found out that the rate of yield of viscosity and the final viscosity are highly dependent on the brine composition, the method of preparation and the activation. According to the amount of residual polymer after degrading, hydroxyethyl cellulose (HEC) was found to be the less damaging polymer, no polymer is as effective as HEC to be used with heavy brines. Nevertheless, HEC cannot viscosify any brine formulation, especially when the density of brine is high, and therefore the activation process is needed. They also concluded that the efficiency of activated HEC is higher than HEC powder and the activated HEC reduces the time of yielding and requires smaller amount of polymer. (Darlington et al., 1982)

The good optimization of particulates size can be an effective way to reduce the fluid loss into the formation. Brans stated that the large solid particles of the fluid loss additives should be at least one-third the diameter of the pores openings of the formation. (Tuttle and Barkman, 1974)

Another advances in the design of the particulate-weighted fluid is using acid-soluble weighting materials, such as calcium carbonate, magnesium carbonate, and itabirite. The resulted filter cake could be easily removed by washing the formation by hydrochloric acid. Calcium carbonate-brine system was found to be the best because calcium

carbonate is available with different particle sizes, chemically stable, inexpensive, and dissolves in HCl. Moreover, some water-soluble polymers could be added to this system to reduce the fluid loss and to improve the fluid suspension capacity. Patton and Phelan reported that itabirite is less soluble in HCl than calcium carbonate but it provides higher density that can reach up to 25 ppg. (Patton and Phelan, 1985)

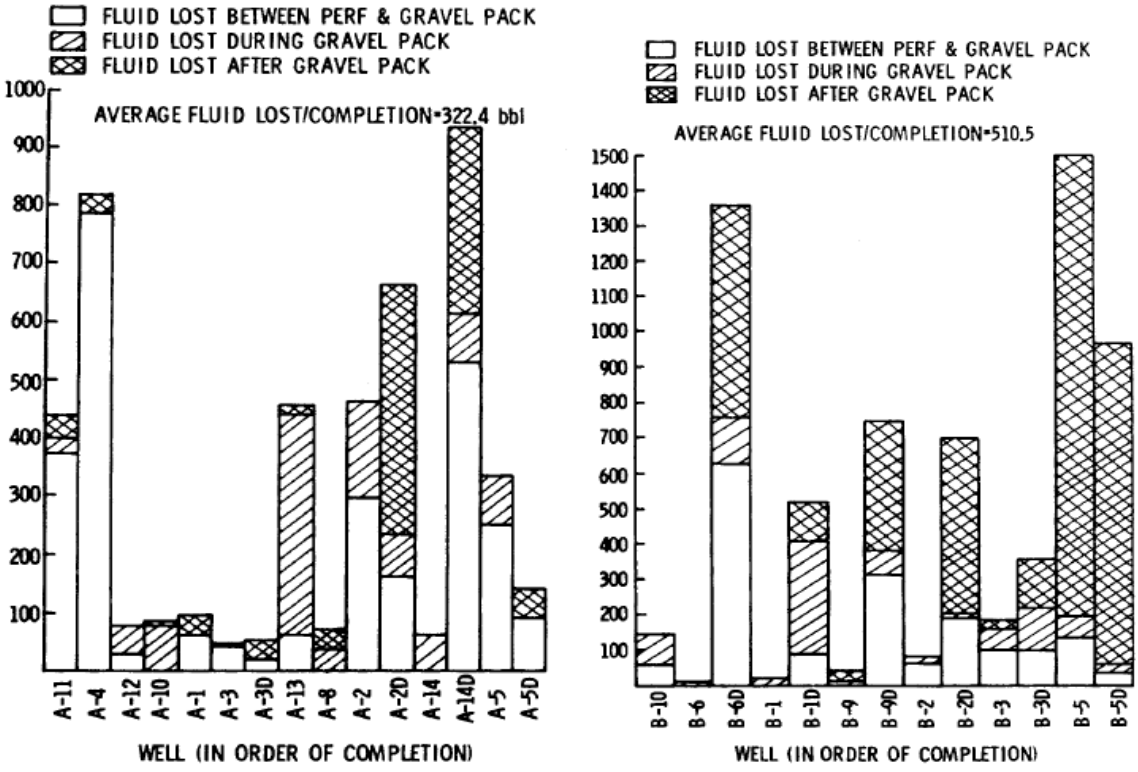

Figure 2.1: Completion Fluid Loss to Formation- Total Fluid Lost per Completion (Spies et al., 1983)

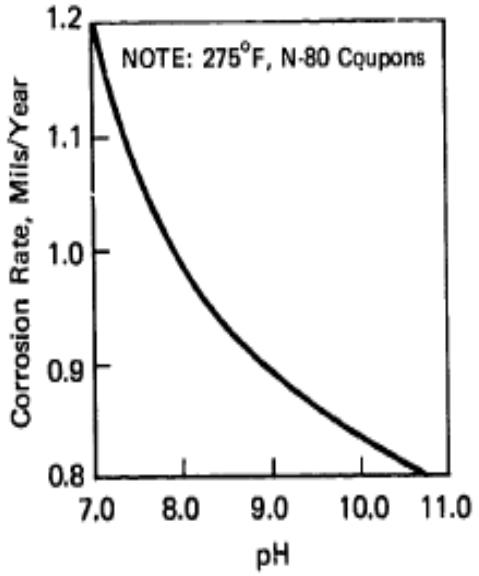

Figure 2.2: Corrosion Rate of CaBr2 Brine versus pH (Paul and Plonka, 1973)

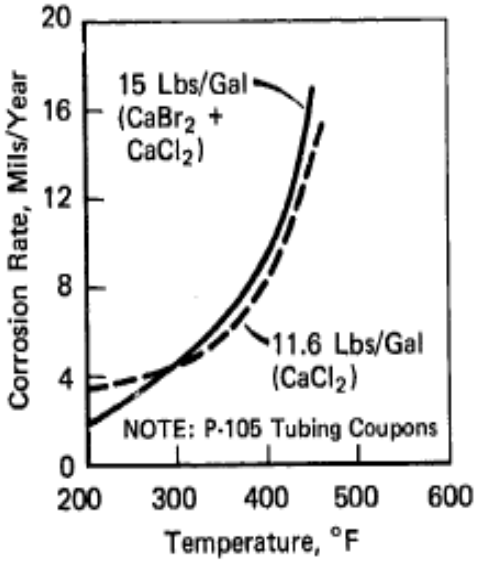

Figure 2.3: Corrosion Rate of Brine versus Temperature (Paul and Plonka, 1973)

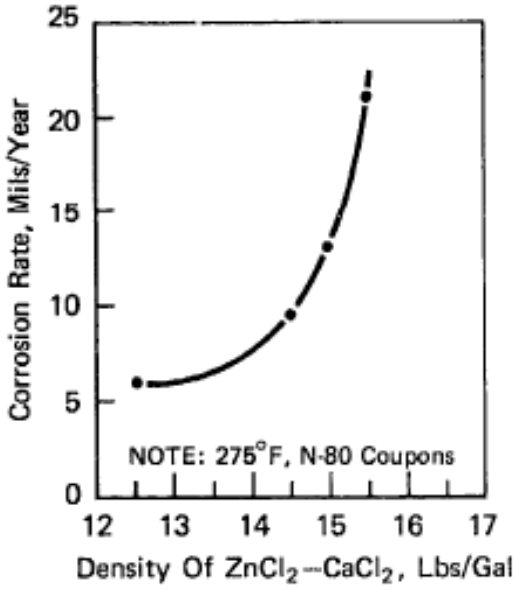

Figure 2.4: Corrosion Rate of ZnCl2-CaCl2 Brine versus Density (Paul and Plonka, 1973)

In 1984, Thomas D. C. et al. pointed out that pressure and temperature have a significant impact on the brine density, a good prediction of the resultant fluid density does help in optimizing the fluid formulation and cost and with poor estimation either a failure in well control would occur in case of low estimates or a high amount of fluid losses due to the excessive overbalance in case of conservative estimates. Accordingly, they developed a model to estimate the brine density- $CaCl_2$, $CaBr_2$, and $ZnBr_2$, accounting for the effect of pressure and temperature. The model was based on the experimental and literature data.(Thomas et al., 1984)

Formate brine is the solution of alkali-metal salts of formic acid. In general, these alkali-metals are: sodium, potassium, and cesium. Cesium formate is the highest density. Water-soluble polymers and filtration control agents should be added to the solution to meet all the needed properties of completion and workover fluids. These formulations were proved to be environmentally friendly and could be reused after treatment. (Downs et al., 1994) Moreover, many cases in the literature have shown a great success of using formate brine as a drill-in and completion fluid. (Berg et al., 2009; Black et al., 1990; Carnegie et al., 2013; Conners and Bruton, 1979; Downs and Fluids, 2011; Downs et al., 2006) Nevertheless, more efforts are needed to control the losses of these costly fluids. (Black et al., 1990)

In 1996, Ramsey et. al. investigated the use of cesium formate (18.7 ppg) as a drill-in fluid. Cesium formate showed higher drilling rate when compared with water-base hematite drilling mud. The formed filter cake was very thin and a high spurt loss was exhibited. (Black et al., 1990)

In 2003, Javora et. al. spotted the light on other problems of using formate brines as a completion and workover fluids by studying the chemistry of formate brines under downhole conditions. They concluded that corrosion and decomposition of these fluids are likely, especially in the presence of hydrogen sulfide and carbon dioxide and this could be mitigated to certain extent by adding buffer additives. They also concluded that the corrosion and decomposition is highly dependent on the metal type that the brine is in contact with. (Javora et al., 2003)

One more problem of using formate brines is that they affect the reading of nuclear logs, due to the high filtrate invasion. (Galford et al., 2005; Moake, 2012) In 2005, in an effort to eliminate the effect of brine-based mud on the reading of nuclear logs, Galford et. al. proposed a model that estimates the additional effect of brine filtrate on log readings. This model was based on laboratory measurements and numerical simulation. (Galford et al., 2005)

2.3 Formation Damage from Completion and Workover Fluids

There are many research and field studies were reported in the literature related to formation damage and many techniques were developed to prevent, mitigate, or remove the damage induced by drilling, completion, or well stimulation fluids.

In 1954, Krueger and Vogel investigated the effect of different types of drilling fluids, oil-base mud, water-base mud, emulsion mud, on several cores of Berea sandstone under high pressure high temperature dynamic conditions (**Table 2.2**). A permanent damage to the cores resulted due to solid particles invasion. They found out that this damage

depends on fluid type and exposure time. The longer the exposure time the higher permeability reduction. They also concluded that the damage caused by fluid filtrate in water-sensitive formations is higher than that caused by solid invasion for a short time of exposure but, for 20 to 30 days of completion the later will be more damaging to the formation (**Figures: 2.5, 2.6 & 2.7**). (Krueger and Vogel, 1954)

In 1974, Tuttle and Barkman conducted a study on the completion fluids that had been commonly used by Shell Company in the Gulf Coast area, which were: filtered or unfiltered bay water with 5% NaCl, and bay water with 5% NaCl with adding guar gum. They found out that bay water solids caused a high decrease in the core permeability (**Figure 2.8**) and only 10% to 30% of the original permeability could be restored by back flowing with brine and not more than 50% could be regained by acid treatment. They also concluded that, in case of guar gum damage, 50% of the original permeability could be regained by back flowing after breaking the guar gum and only 25% could be regained with unbroken guar gum (**Figures: 2.9 & 2.10**). (Tuttle and Barkman, 1974)

Table 2.2: Average Properties of Field Drilling Fluids Used for Tests (Krueger and Vogel, 1954)

Drilling Fluid	Weight, PCF	Marsh Funnel Viscosity, Sec at 170 °F	Fluid Loss (API), cc				Time in Use in Drilling Well, days
			Room Temperature		200 °F		
			15 min	1 hour	15 min	1 hour	
Emulsion	68	45	0.8	3.2	5	9.5	27+
Oil-base							
Sample1	65	60	-	-	-	-	4.5
Sample2	63	54	-	-	-	-	11
Clay-water							
Sample1	73	50	11	11	9	20	2.5
Sample2	74	45	3.2	7.8	6	14	27

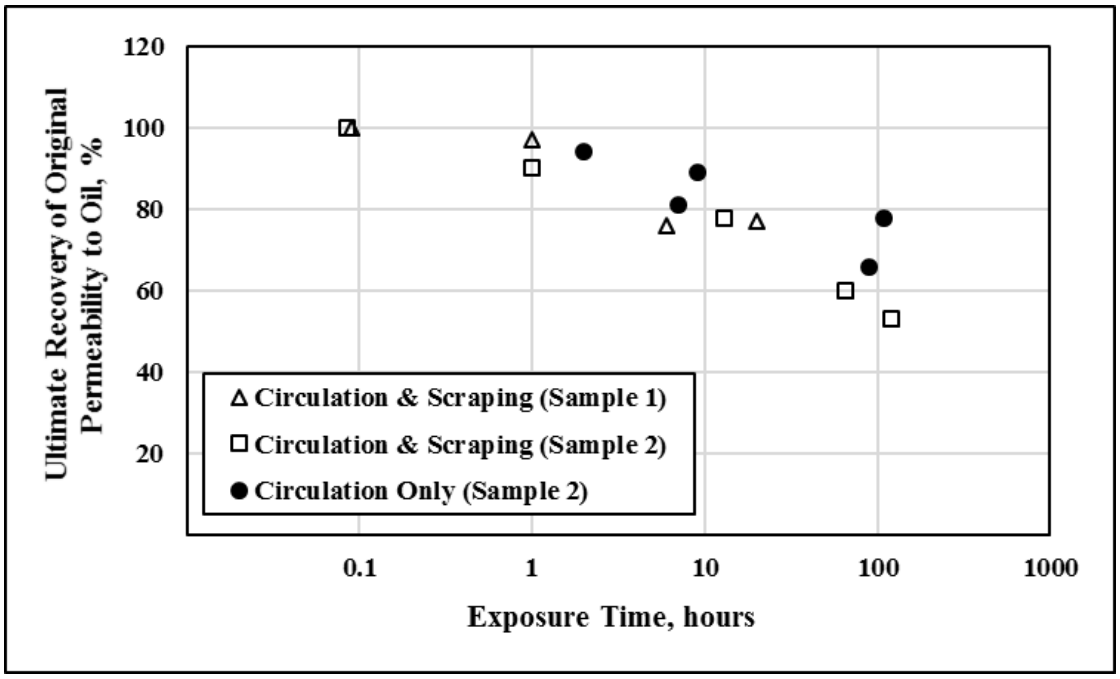

Figure 2.5: The Effect of Field Oil-Based Mud on Permeability to Oil of 1-in. Diameter Berea Sandstone Cores (Krueger and Vogel, 1954)

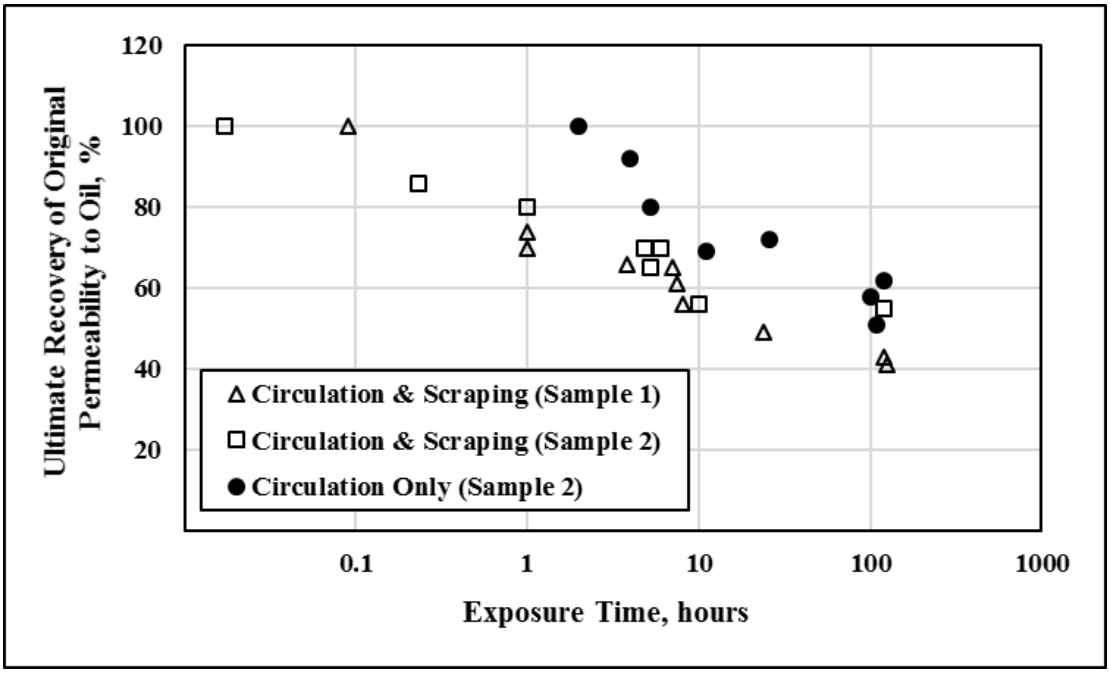

Figure 2.6: The Effect of Field Clay-Water Mud on Permeability to Oil of 1-in. Diameter Berea Sandstone Cores(Krueger and Vogel, 1954)

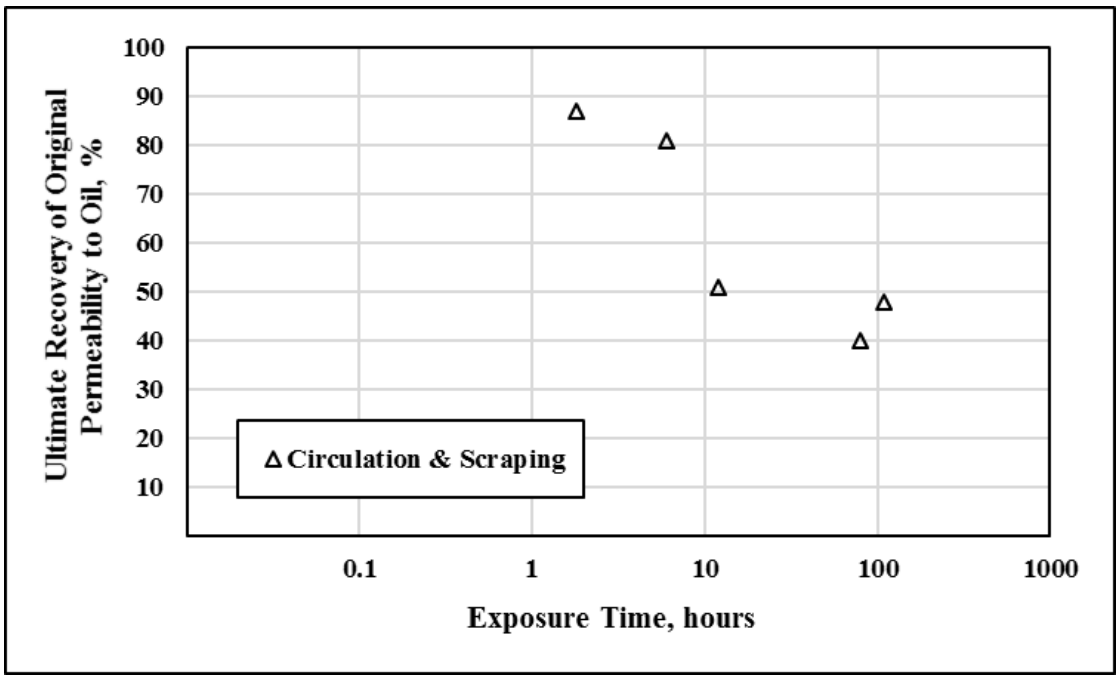

Figure 2.7: The Effect of Field Emulsion Mud on Permeability to Oil of 1-in. Diameter Berea Sandstone Cores
(Krueger and Vogel, 1954)

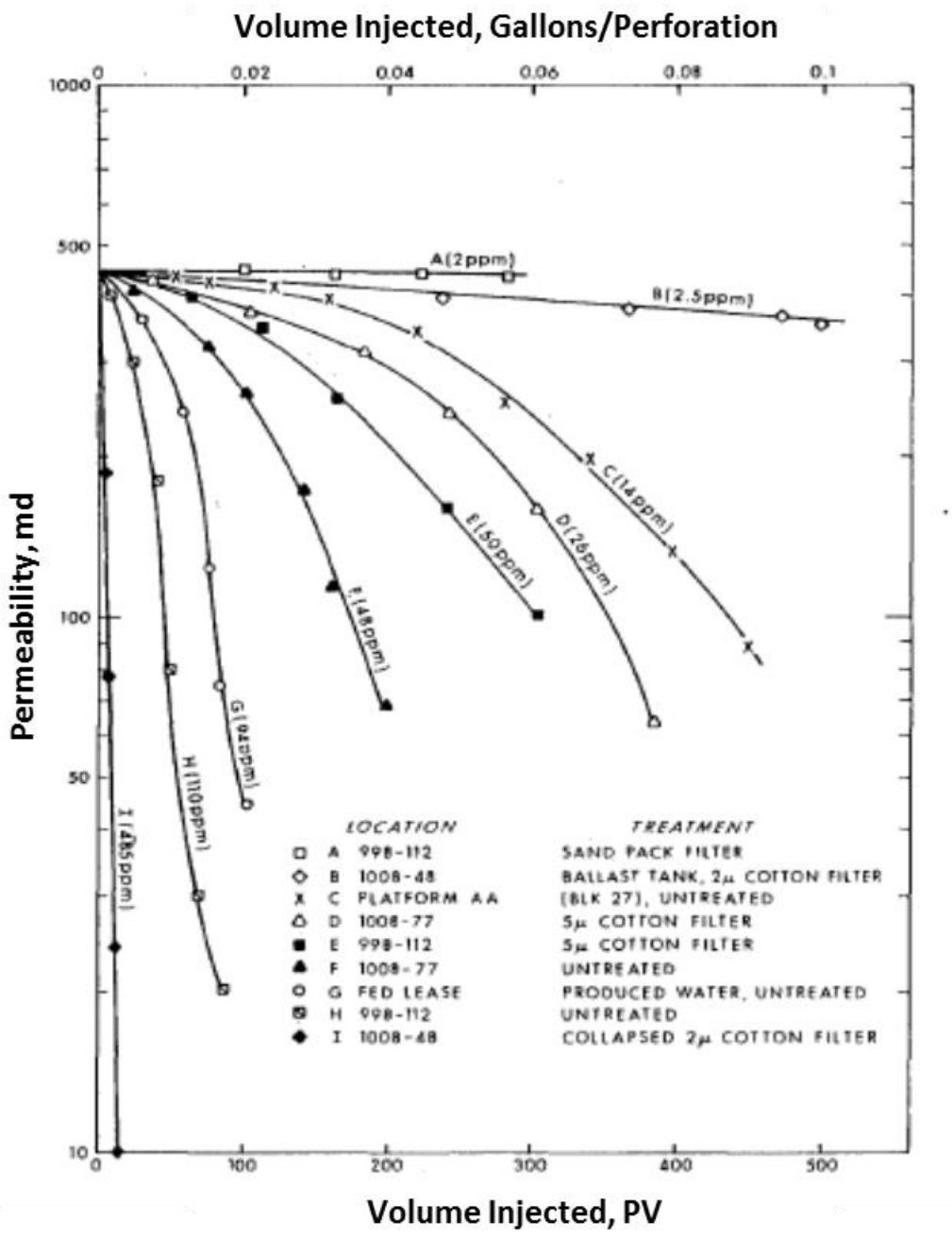

Figure 2.8: Apparent Permeability Reduction in Cypress Sandstone Cores due to Injection of Various Treated and Untreated Bay Water (Tuttle and Barkman, 1974)

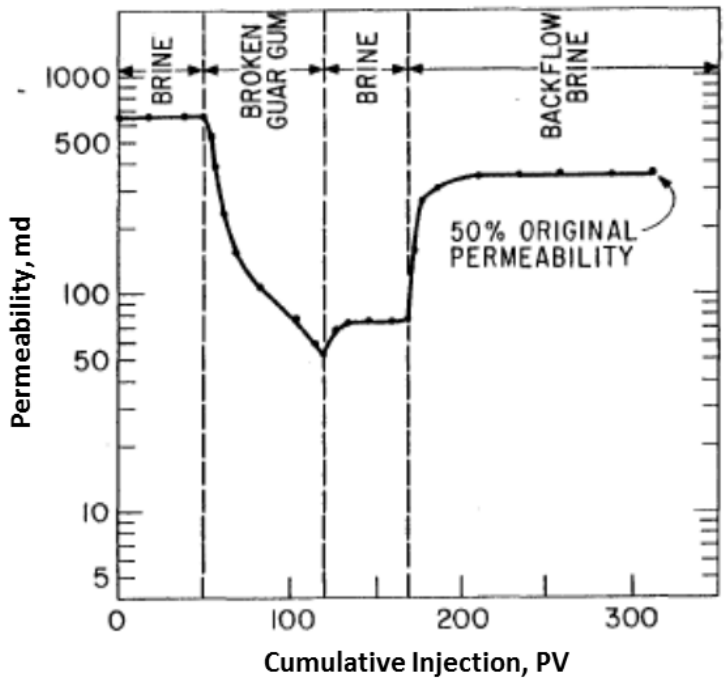

Figure 2.9: Effect of Enzyme-Broken Guar Gum Solution on Permeability of Cypress Sandstone (Tuttle and Barkman, 1974)

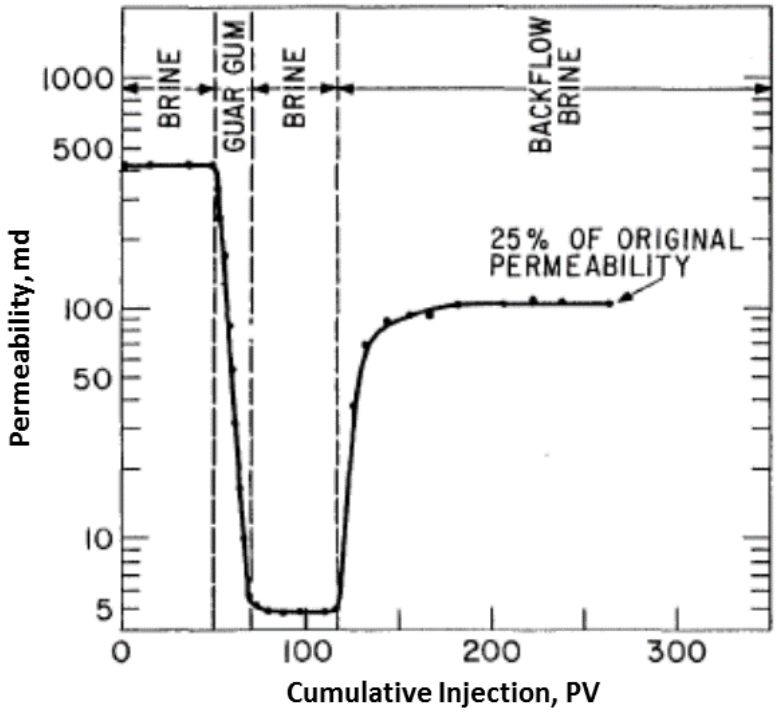

Figure 2.10: Effect of Unbroken Guar Gum Solution on Permeability of Cypress Sandstone (Tuttle and Barkman, 1974)

In 1985, Morgenthaler L. N. conducted a study on sandstone cores to investigate the formation damage caused by high-density brine completion fluids. He reported that the main causes of formation damage when using solids-free completion fluids, especially calcium carbonate, is saults precipitation not because of the unfavorable interactions between the fluid filtrate and the clay that presents in the cores. Formulating the brines that have a density greater than 14 ppg with at least 8% of zinc bromide was recommended to decrease the pH in order to prevent saults precipitation. (Morgenthaler, 1986)

In 2001, Suri and Sharma stated that a proper selection of particle size would significantly minimize the formation damage caused by solid particles from drilling and completion fluids. They also developed a filtration model that quantifies the formation damage caused by different particle sizes. This model yielded reasonable results when it was compared with actual experimental data. (Suri and Sharma, 2001)

2.4 Laboratory Evaluation for Completion and Workover Fluids

Evaluating completion and workover fluids requires a series of experiments and measurements that should be conducted prior to the field applications under the expected downhole conditions in order to simulate the actual case. In general, these measurements include rheological properties measurements and fluid stability and compatibility with the target formation.

From field experience, some compatibility issues are well documented in the literature and do not require performing any experiments, for instance, active clays cannot be drilled using non-inhibited water-based mud. Nevertheless, usually compatibility study should be conducted especially with new fluid formulations or in critical conditions.

In 1971, Eaton and Smithey modified the conventional core flooding apparatus to quantify the return permeability and the permeability reduction that resulted from different completion and workover fluids. To simulate the actual fluid invasion, a new line was added to inject the tested fluid in the reverse direction of the core. Flow rate was recorded to measure the permeability before and after injection of the tested fluid. (Eaton and Smithey, 1971) **Figure 2.11** shows a schematic illustration of return permeability test apparatus.

The return permeability is then calculated using **equation (2.1)**:

$$\% \text{ Return K} = \frac{\text{K at any point of return flow}}{\text{K original}} \text{x100} \quad \ldots\ldots\ldots\ldots (2.1)$$

2.5 Barite Removal

Barite is one of the most common used weighting materials for conventional drilling fluids. It has been used since 1920s due to its high density, 4.5 g/cm^3, which makes it a good candidate to control high pressure high temperature wells. Moreover, it is easy to handle and relatively inexpensive. Since, barite is used for well control, formation damage is highly anticipated due to solids invasion. In fact, barite has a very low solubility in water therefore, many barite dissolvers were introduced to eliminate and

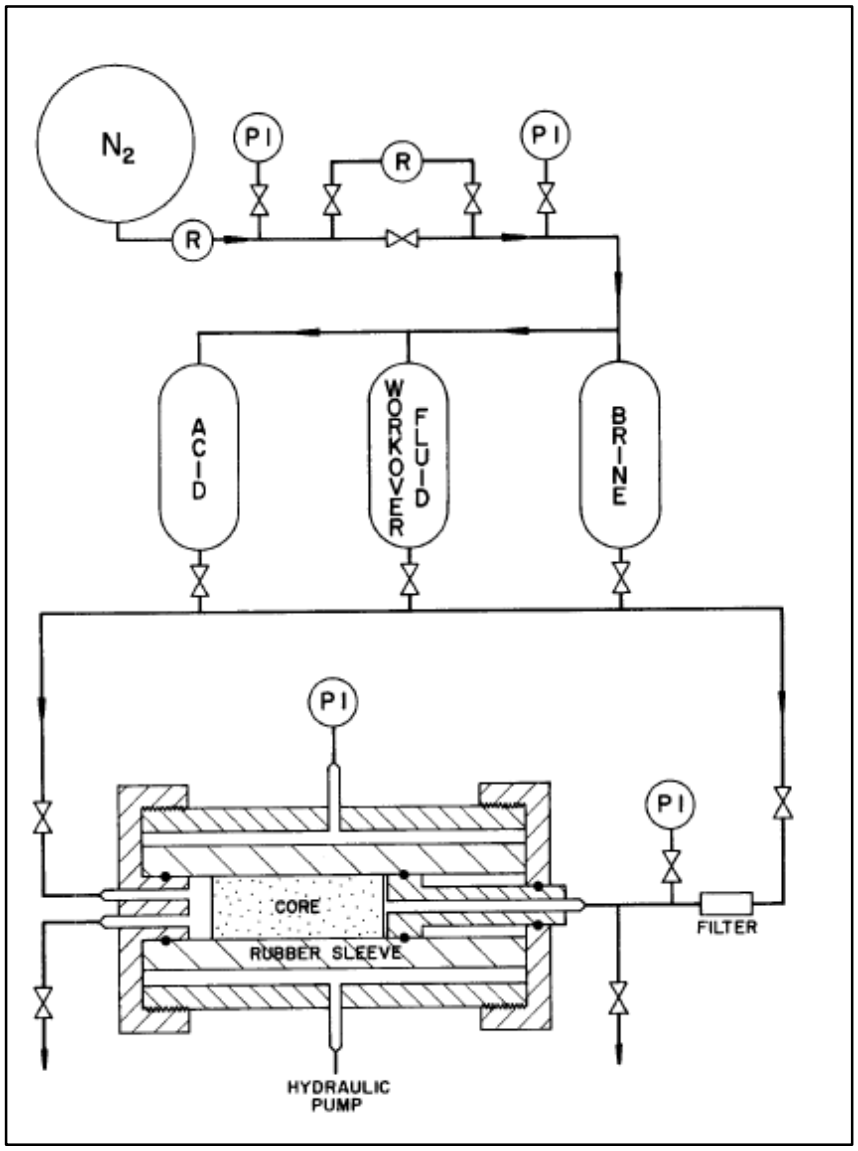

Figure 2.11: Schematic of Return Permeability Test Apparatus (Eaton and Smithey, 1971)

minimize the induced damage. Most of these dissolvers are based on chelating agents. (Clemmit et al., 1985; Film and Process, 1977; Frenier, 2001; Lakatos et al., 2002; Mallion et al., 1972; Nasr-El-Din et al., 2004; Paul and Fieler, 1992; Putnis et al., 1996; Richard and James, 1990; Wang et al., 1999)

In this study, a newly developed formulation that was proposed by Ba Geri et al. (2016) was used to investigate the effect of barite particle size on barite removal. They studied

the performance of different chelating agents on barite removal after optimizing the concentration of each chelation agents. Afterwards, many catalysts were tested with different concentrations and the final formulation gave a solubility of around 41 g/L at 150 °C for 24 hours.

Table 2.3: Completion and Workover Fluids Selection Guide (Millhone, 1983)

Fluid	Density, ppg	Stability (Static)	Clay Reactions	Solids	Corrosion
Gas:					
Air/natural gas	0 to 8.3	unlimited	-	-	minor
Mist	0 to 8.4	none	minor	minor	variable
Foam	0 to 8.5	limited	minor	-	variable
Methanol	6.6	unlimited	minor	-	variable
Oil:					
Diesel	7.03	very long	-	-	-
Crude(treated)	7 to 8	very long	-	-	-
Emulsions	7 to 8.3	long	minor	-	minor
Weighted oil	7 to 17	variable	-	Calcite, siderite	-
Weighted emulsions	8.3 to 17	long	minor	Calcite, siderite	minor
Water:					
Fresh	8.3	unlimited	none to extreme	-	variable
Seawater (treated)	8.5	very long	none to extreme	-	minor
Brines:					
KCl	8.3 to 9.8	very long	none to major	-	minor
NaCl	8.3 to 10	very long	none to major	-	minor
CaCl2	8.3 to 11.6	very long	none to major	-	minor
CaBr2	8.3 to 15.2	very long	none to major	-	moderate
ZnBr2	8.3 to 19.2	very long	none to moderate	-	major
Weighted Water/Brine:					
Salt	8.3 to 15	short to very long	none to major	NaCl	minor
Carbonates	8.3 to 17	short to very long	variable	Calcite, siderite	variable

2.6 Barite Ball Milling

Ball milling is a process that basically used to grind and blend materials where particle size is reduced to the desired size. A ball mill works on the principal of impact and attrition where balls are the grinding media that utilizes the energy of rotation and vibration. In general, there are two techniques of grinding either wet or dry grinding and both have their own applications. However, optimizing the operating parameters such as,

milling time, balls-to-powder ratio, balls specifications, and rotational speed- RPM, would determine the average particle size of the produced materials, therefore, an experimental study should be conducted prior to production.

In fact, many studies on ball milling of different materials have been conducted and well documented in the literature. (Deniz, 2012; Hamel et al., 2012; Khalil et al., 2010; Patel et al., 2012; Teke et al., 2002; Umucu et al., 2014) **Table 2.4** shows some of the studies that have been conducted to produce barite Nano particle.

Table 2.4: Barite Ball Milling Case Studies

No.	Paper	Materials	Equipment	Time interval	Range of parameters
1	Chetan M. Patel, Z.V.P. Murthy , Mousumi Chakraborty Effects of operating parameters on the production of barium sulfate nanoparticles in stirred media mill 2012	barite (11.18 microns) Dispersant: sodium salt of polyacrylic acid (20 wt%) dioinized water for suspension HNO3 and Naoh for ph control ph=6 cesium oxide-stabilized zirconia grinding beads	wet grinding using stirred media mill stainless steel vessel (0.736 mL)	30 min sample: 2-3 mL	dispersant concentration grinding time ph control pin length media diameter solid mass fraction stirrer speed 1200 RPM
2	E. Teke, M. Yekeler *, U. Ulusoy, M. Canbazoglu Kinetics of dry grinding of industrial minerals: calcite and barite 2002	barite and calcite barite: -800, +300 micometers (various feeds)	ball milling dry grinding		ball size: 46, 26, 12.8 mm ball specific gravity:7.81 g/cc powder-ball ratio: 0.5
3	Claudine Hamel, Thierry Brousse, Daniel B´elanger, and Daniel Guay Effect of Ball-Milling on the Physical and Electrochemical Properties of PbO2 and PbO2/BaSO4 Nanocomposite 2012	PbO2 and BaSO4 (5, 10 and 20w% BaSO4)	ball milling SPEX 8000 mill stainless steel crucible and balls	15 min to 45 hrs	3 balls balls sizes: 2of 7/16", 1 of 1/2" constant ball-to-powder ratio: 5 to 1 final size: 10 nm
4	Hazem Khalil et. al BaSO4-, CaF2-, and Al-Maleate-Derived Nanocomposite Coatings with Excellent Mechanical, Thermal, and Optical Properties 2010	BaSO4 30wt%+ CaF2 (nano composite)	1- planetary ball mill Pulverisette 5 balls of 0.5–0.8cm	1, 2 hrs	speed: 400 rpm
			2- high-energy attrition mill ceramic beads (ZrO2) 0.7–1.2mm	30 min	speed: 3000 rpm
5	Vedat Deniz The effects of ball filling and ball diameter on kinetic breakage parameters of barite powder 2012	barite (-0.85, +0.106 mm)	ball milling dry milling cell: 6280 cc		speed: 76 rpm ball size: 15, 25.4, 40 mm ball specific gravity:7.8 g/cc ball filling volume: 25, 35, 45

CHAPTER III

RESEARCH OBJECTIVES AND METHODOLOGY

3.1 Problem Statement

The complexity and the critical downhole conditions that will be encountered while completing high pressure high temperature wells require a special formulation of completion and workover fluids to ensure a safe and successful operation. The introduced fluid should provide enough hydrostatic pressure to equalize the high formation pressure and should be stable under that critical downhole conditions.

Moreover, since oil or gas formation, rig personnel, and environment are all exposed to completion and workover fluids, these formulations should not have an impact on the environment and well productivity which will consequently decrease the ultimate oil or gas production and the feasibility of the completion and workover operations.

In fact, many fluids are used in the industry for completion and workover operations at high pressure and high temperature conditions. However, some of the proposed fluids have environmental or technical issues while others are costly.

Conventional drilling fluids have many good characteristics such as, they have high density and they are easy to handle, environmentally friendly, and relatively inexpensive. All these characteristics lead us to reconsider barite as a weighting material for completion and workover fluids in HPHT wells after solving the associated problems, formation damage and fluids stability.

3.2 Research Objectives

This work aims to:

1. Study the effect of the average particle size on barite removal using solubility test.
2. Study the effect of reducing barite particle size on fluids stability under HPHT conditions.
3. Design a new formulation of completion fluids that could minimize the formation damage that might result from solids invasion using micronized barite with appropriate additives.
4. Evaluate micronized barite as a completion fluid weighting material to complete HPHT wells.

3.3 Research Methodology and Experimental Work

3.3.1 Materials

A sample of commercial barite (obtained from a local supplier) with an average particle size of 35 microns was used in this study. The particle size distribution and the elemental composition of barite are shown on **Figures: 3.1 & 3.2**. DTPA chelating agent of initial concentration of 40 wt% was used. DTPA was diluted with deionized water to obtain the desired concentration of 20 wt%. Potassium carbonate was added to DTPA with a concentration of 6 wt% as a catalyst to remove barite. Several barite samples with different particle sizes were prepared using sieve analysis and ball milling machine. NaOH and HCl were used to adjust the pH of deionized water for zeta potential measurements. Some additives (obtained from local supplier) were used to formulate the

completion fluid and they are listed in **Table 3.1**. Ceramic disks of 2.5-inch diameter and 0.25-inch thickness were also used in filtration test.

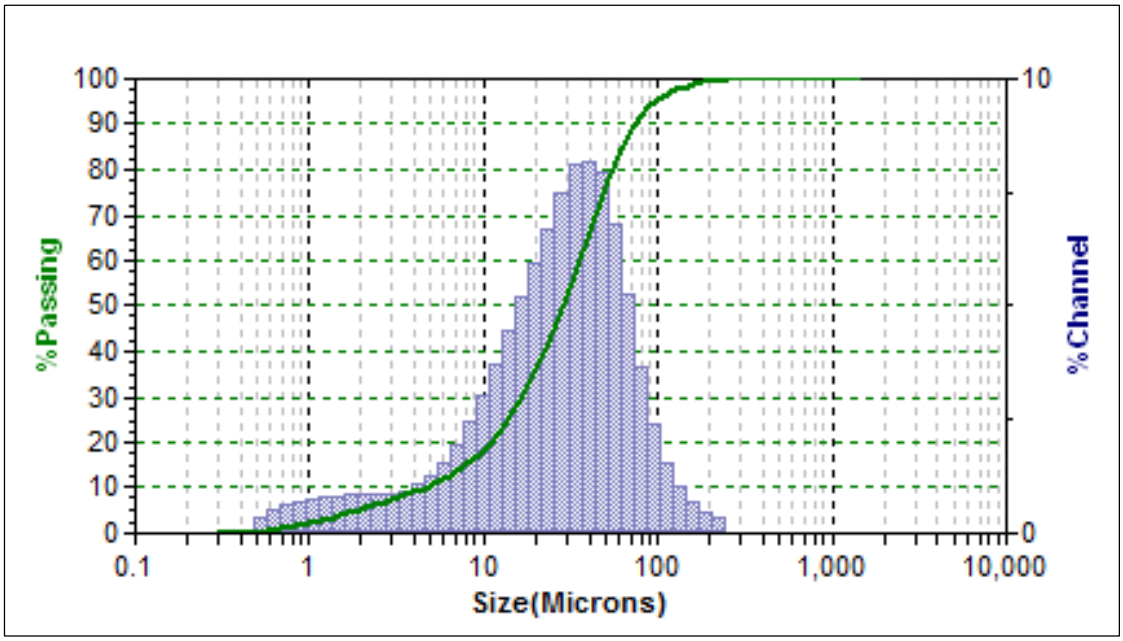

Figure 3.1: Particle Size Distribution of Raw Barite Sample

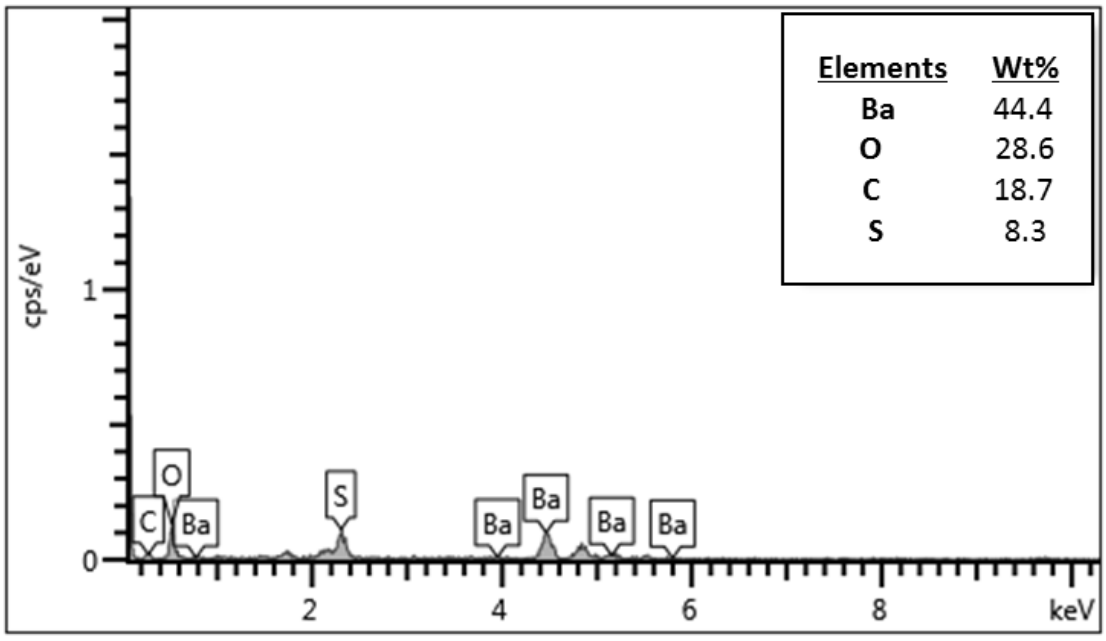

Figure 3.2: Elemental Composition of Raw Barite Sample

Table 3.1: Completion Fluid Additives

Additive	Specific Gravity	Function
Soda ash	2.54	Maintain calcium level
Bentonite	4.3	Viscosifier
Xhanthan polymer	0.801	Viscosifier
Potassium chloride, KCl	1.98	Clay stabilizer
Potassium hydroxide, KOH	2.12	pH adjustment
Calcium carbonate, $CaCO_3$	2.71	Bridging agent
Barite	4.5	Weighting material
Sodium sulfite	2.63	Oxygen scavenger

3.3.2 Barite Ball Milling

Ball milling was mainly used to reduce the average particle size of barite to investigate the effect of particle size reduction on fluid stability as well as its impact on enhancing barite removal. TENCAN planetary ball milling machine (**Figure 3.3**) was used to reduce barite particle size. From the previous studies, an initial guess to all the operating parameters was used and kept fixed throughout the experiments and the milling time was the only variable parameter to be optimized. These parameters are: feed weight, balls-to-powder ratio, balls size, rotational speed, and milling time.

A 40-gram sample of barite that has a particle size less than 75 microns (with an average particle size of 35 microns) was used as the feed to ball milling machine. Stainless steel balls of 7.93 mm diameter were used as grinding media with balls-to-powder ratio of 10. The machine was running at 500 RPM rotational speed. The experiment was run for 10 hours milling time. 5-gram samples were taken after 0.5, 1, 2, 3, 5, 8, and 10 hours while, for consistency, the balls-to-powder ratio was kept constant at 10 during the whole experiment. Afterwards, these samples were characterized using scanning electron microscope, SEM (**Figure 3.4**) and X-Ray diffraction, XRD (**Figure 3.5**) techniques, in

addition to the particle size analysis, using TURBOTRAC particle size analyzer (**Figure 3.6),** in order to investigate the effect of milling time on the produced barite. **Table 3.2** summarizes the operating parameters of the ball milling experiment.

Table 3.2: Ball Milling Operating Parameters

Parameter	Description
Barite Feed	35 microns, 40 grams
Balls-To-Powder Ratio	10
Balls Size	7.93 mm
Balls Weight	2.05 grams
Rotational Speed	500 RPM
Milling Time	10 hours
Samples Intervals	0, 0.5, 1, 2, 3, 5, 8, 10 hours
Sample weight	5 grams

Figure 3.3: TENCAN Planetary Ball Milling Machine

Figure 3.4: SEM Machine

Figure 3.5: XRD Machine, D8 Advance

Figure 3.6: TURBOTRAC Particle Size Analyzer

3.3.3 Solubility Test

Solubility tests were conducted to measure the amount of barite solid particles that could be removed, i.e. it gives an indication to the amount of permeability that could be restored. A new formulation that has been introduced by Ba Geri et. al. (2016) was used to investigate the effect of particle size on barite removal.

DTPA at high pH with a concentration of 40 wt% was diluted to 20 wt% using deionized water. 6 wt.% of potassium carbonate was added to the solution as a catalyst. Different samples of barite with different particle size were added to the solution, with a ratio of 1 gram to 20 ml, and put on a multiple heating magnetic stirrer at dynamic condition for 24 hours at a temperature of 300 °F (**Figure 3.7**). After 24 hours the samples were filtered using filter papers to quantify barite solubility (**Figure 3.8**). Afterwards, the filtered solids were left in the oven for 2-3 hours to make sure that they were completely dry. The weight of filter papers was measured before and after the test using weight balance

(**Figure 3.9**). Then, the solubility was calculated using **equation (3.1) & (3.2)**. **Table 3.3** summarizes solubility test experiments.

$$\text{Solubility}, \% = \frac{W_{Solids} - (W_{after} - W_{before})}{W_{Solids}} \text{x}100 \ldots\ldots\ldots\ldots\ldots\ldots (3.1)$$

$$\text{Solubility}, g/L = \frac{W_{Solids} - (W_{after} - W_{before})}{V_{Solution}} \text{x}1000 \ldots\ldots\ldots\ldots\ldots\ldots (3.2)$$

Where;

W_{solids}= Weight of barite solid particles, g

W_{after}= Weight of the filter paper after the test, g

W_{before}= Weight of the filter paper before the test, g

$V_{solution}$= Volume of the solution, mL

Table 3.3: Solubility Test Summary

Parameter	**Description**
Barite	1 gram
DTPA	20 wt.%
Potassium Carbonate	6 wt.%
Total Solution	20 mL
pH	13.5
Temperature	300 °F
Condition	Dynamic
Solubility Time	24 hours
Drying Time	2-3 hours

Figure 3.7: Solubility Test Setup

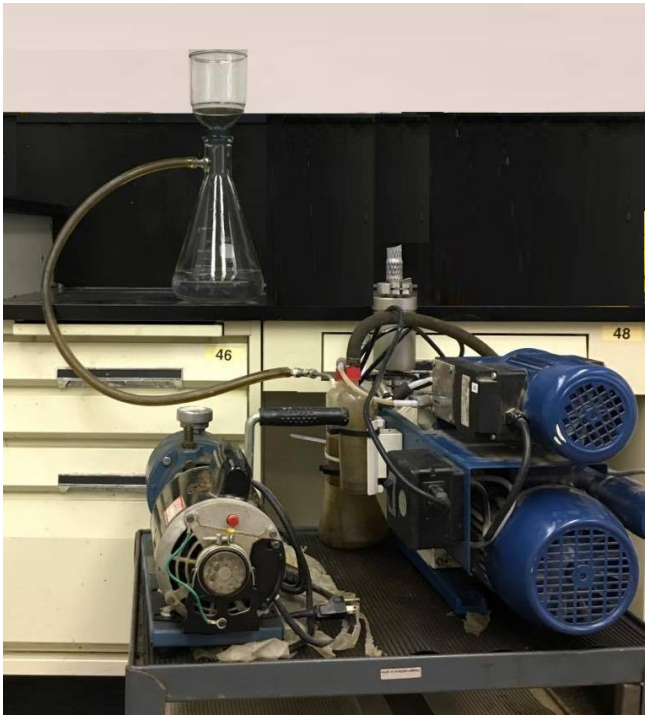

Figure 3.8: Filtration Setup

Figure 3.9: Weight Balance

3.3.4 Zeta Potential Measurements

Zeta potential is the physical property of solid particles in suspension and it is a measure of charge, repulsion or attraction, between colloids. Zeta potential was measured over a wide range of pH to investigate the effect of barite particle size on fluid stability, a fluid that has a zeta potential value in the range from 0 to ±30 is considered unstable (**Table 3.4**).

Several samples of water were prepared with different pH, alkaline, neutral and acidic. Caustic soda and HCl were used as pH control agents. Different barite samples with different particle sizes were added to water samples with a concentration of 0.5 wt.%. Then these samples were placed in a multi-wrist shaker at room temperature for 20 to 24 hours to ensure that the charges equilibrium took place (**Figure 3.11**).

Afterwards, a sample from each solution was taken using a syringe with 5 µm filter. Zeta potential was measured using *ZetaPALS* zeta potential analyzer (**Figure 3.12**). An

average value of zeta potential was calculated from 5 readings and 20 runs for each reading.

Table 3.4: Zeta Potential and Colloids Stability

Zeta Potential [mV]	Stability Behavior of the Colloid
0 to ±5	Rapid coagulation or flocculation
±10 to ±30	Incipient instability
±30 to ±40	Moderate stability
±40 to ±60	Good stability
More than ±61	Excellent stability

Figure 3.10: JENWAY pH Meter

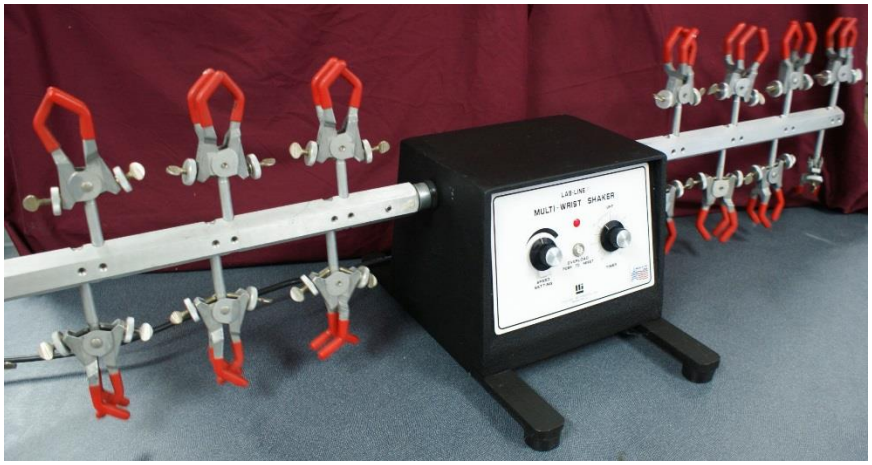

Figure 3.11: LAB-LINE Multi-Wrist Shaker

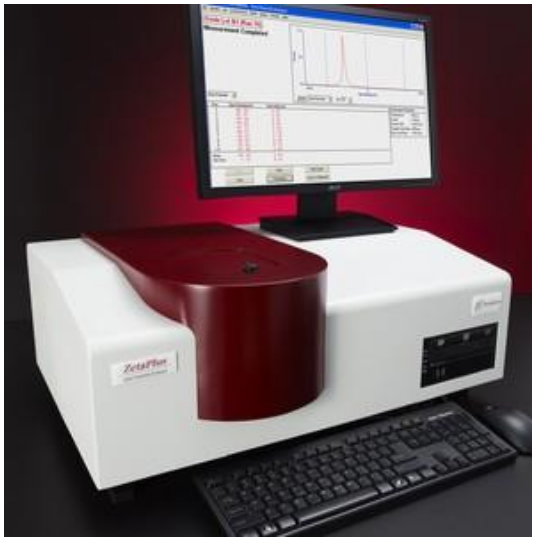

Figure 3.12: ZetaPALS Zeta Potential Analyzer

3.3.5 Fluid Preparation and Rheology Measurements

A high density completion fluid, 15.3 ppg, was prepared. Water was added with a ratio of 0.7 bbl, (245 g in lab units), and poured in the drilling fluid mixer (**Figure 3.13**). Defoamer and soda ash were added to prevent the formation of foam and to maintain the level of calcium in water, respectively. Then, other additives were added and mixed for some time, starting by viscosity control agent, Xanthan polymer and bentonite, starting by a low rotational speed and then the rotational speed was increased gradually as viscosity built up. Different sizes of barite were added as weighting material. **Table 3.5** lists all the additives, their ratios and functions, and the mixing time.

After fluid preparation, fluid density was measured using mud balance (**Figure 3.14**), and all the rheological properties were measured at 120 °F using *Fan35A* viscometer (**Figure 3.15**). Rheology measurements were also conducted at HPHT conditions using *GRACE m5600* rheometer (**Figure 3.16**) to investigate fluid stability at that critical conditions. All fluid properties are listed in **Table 3.6**.

Table 3.5: Completion Fluid Formulation

Name	Description	Field Units/ Lab Units	Mixing Time, min	Function
Water	0.7	bbl/g	-	Base
Defoamer			-	Anti-Foam Agent
Soda ash	0.5	lb/g	10	Maintain Calcium Level
KOH	0.5	lb/g	10	pH Adjustment
Bentonite	4	lb/g	10	Viscosifier
XC-polymer	1-1.5	lb/g	20	Viscosifier
KCl	20	lb/g	10	Clay Stabilizer
CaCO3	5	lb/g	10	Bridging Agent
Barite	350	lb/g	10	Weighting Material
Sodium sulfite	0.25	lb/g	10	Oxygen Scavenger

Table 3.6: Completion Fluid Properties

Property	Unit	Range
Density	pcf	115
Plastic Viscosity	cP	20-30
Yield Point	Lb/100 ft^2	20-25
Gel Strength, 10 sec	Lb/100 ft^2	10
Gel Strength, 10 min	Lb/100 ft^2	15
Gel Strength, 30 min	Lb/100 ft^2	20
pH	-	9.5-10

Figure 3.13: Drilling Fluid Mixer

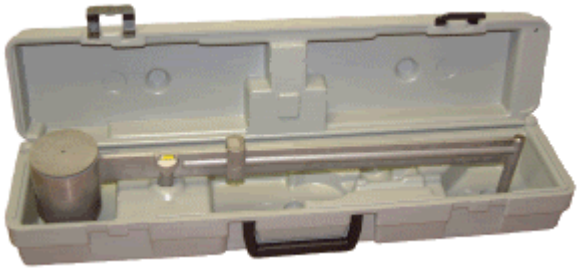

Figure 3.14: Mud Balance

Figure 3.15: Fan 35A Viscometer

Figure 3.16: GRACE m5600 Rheometer

3.3.6 Sag Test

Sag test was conducted to measure barite sag tendency to ensure that completion fluids are stable under HPHT conditions. First, completion fluid was agitated using drilling fluid mixer. A Teflon vessel was used to contain the fluid and an aging cell was used to keep the fluid pressurized (**Figure 3.17**). A pressure of 500 psi was applied using nitrogen to prevent fluid from evaporation and the aging cell was put vertically in the oven for 24 hours at different temperatures. The test was conducted at static conditions. After 24 hours, the cell was left to cool down and the pressure was released. Afterwards, two fluid samples were taken from the top and the bottom of the vessel. The densities of the two samples were measured and the sag factor was calculated using **equation (3.3)**. The fluid that has a sag factor between 0.5 and 0.53 would have a good sag performance while a sag factor greater than 0.53 would result in a bad sag performance and solid particles settlement is very likely to occur.

$$\text{Sag Factor} = \frac{\rho_{Bottom}}{\rho_{Bottom} + \rho_{Top}} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3.3)$$

Where;

ρ_{Bottom} = Bottom density, PCF

ρ_{Top} = Top density, PCF

Figure 3.17: Static Sag Test Setup

3.3.7 HPHT Filtration Test

Filtration test was conducted to evaluate the new formulation of completion fluid by measuring fluid loss into the formation. A ceramic disc, of 2.5" diameter and 0.5" thickness, was immersed in fresh water and left for some time to be completely saturated. The ceramic disc was weighted before and after saturation and then placed into the filtration cell then, 350 ml of completion fluid was poured inside the cell. The cell was closed and a pressure of 300 psi was applied using nitrogen cell and the filtration cell was heated gradually up to 250 °F. The test was conducted at static condition.

Afterwards, the valve was opened and the filtrate volume was recorded with time. The experiment was run for 30 minutes then, the valve was closed, the cell was cooled down and the pressure was released. Furthermore, the disc was taken out and the filter cake was characterized by measuring the weight and the thickness. Another part of the experiment was to quantify filter cake removal efficiency. This part was conducted in one step at the same conditions of pressure and temperature using a high pH solution of DTPA (20

wt.%) with potassium carbonate (6 wt.%) and enzyme (7 wt.%) to break the polymer for 24 hours. Eventually, the disc was weighted after the removal. Filter cake removal efficiency was calculated using **equations: (3.4) & (3.5).**

$$\text{Removal Efficiency}, \% = \frac{W_{Filter\ cake} - (W_{d3} - W_{d1})}{W_{\text{Filter cake}}} x100 \ldots\ldots\ldots\ldots\ldots\ldots (3.4)$$

$$W_{\text{Filter cake}} = (W_{d2} - W_{d1}) \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3.5)$$

Where;

$W_{\text{Filter cake}}$= Weight of the filter cake, g

W_{d1}= Weight of the saturated ceramic disc, g

W_{d2}= Weight of the ceramic disc after filtration, g

W_{d3}= Weight of the ceramic disc after removal, g

Figure 3.18: HPHT Filter press, 500 ml, OFITE

CHAPTER IV

RESULTS AND DISCUSSION

4.1 Barite Ball Milling

Ball milling was mainly used to reduce the average particle size of barite to investigate the effect of particle size reduction on fluid stability as well as to see its impact on enhancing barite removal. *TENCAN* planetary ball milling machine was used to reduce barite particle size. A preliminary study was conducted to optimize the milling time and to study the effect of ball milling on barite. From the previous studies in the literature, an initial guess to all the operating parameters was opted and kept fixed and the milling time was the only variable parameter to be optimized. These parameters are: feed weight, balls-to-powder ratio, balls size, rotational speed, and milling time.

Two 40-gram samples of barite that have a particle size less than 75 microns (with an average particle size of 35 microns) were loaded in 2 vials to keep the machine balanced. Stainless steel balls of 7.93 mm diameter were used as grinding media with balls-to-powder ratio of 10. The machine was running at 500 RPM rotational speed. The experiment was run for 10 hours milling time. 5-gram samples were taken after 0.5, 1, 2, 3, 5, 8, and 10 hours while, for consistency, the balls-to-powder ratio was kept constant at 10 during the whole experiment.

Afterwards, these samples were characterized using X-Ray diffraction (XRD), scanning electron microscope (SEM) techniques, in addition to the particle size analysis in order to investigate the effect of milling time on the produced barite.

Figure 4.1 shows the XRD patterns for barite before and after ball milling for 0.5, 1, 2, 3, 5, 8, and 10 hours. This figure shows a series of diffraction peaks that correspond to barite, their full width at half maximum, FWHM, increased as a result of ball milling. No new diffraction peak was observed which indicates that no Fe contamination took place, and the elemental composition from EDS Technique confirmed that.

Barite samples were also characterized using scanning electron microscopy, SEM, to investigate the effect of ball milling on morphological properties. **Figure 4.2** shows the SEM micrographs of barite as received, ball milled barite after 0.5, 1, and 8 hours. A variety in particle size and shape of barite as received sample was observed and after ball milling a very fine powder was produced. As a result of ball milling, small particles clustered and some agglomeration took place due to the fact that large particles breakage depleted oxygen at the surface and consequently increased the surface energy of the small particles that agglomerated to reduce their high energy(Hamel et al., 2012; Rochefort and Guay, 2005) (**Figure 4.3**).

Particle size analysis was performed using *Turbotrac* particle size analyzer. Particle size analysis results endorsed SEM observation and barite particle size was reduced to a few microns after milling for half an hour and then particle size started to increase again due to the small particles agglomeration. **Table 4.1** shows particle size analysis results for barite as received and ball milled after 0.5, 1, 3, and 5 hours.

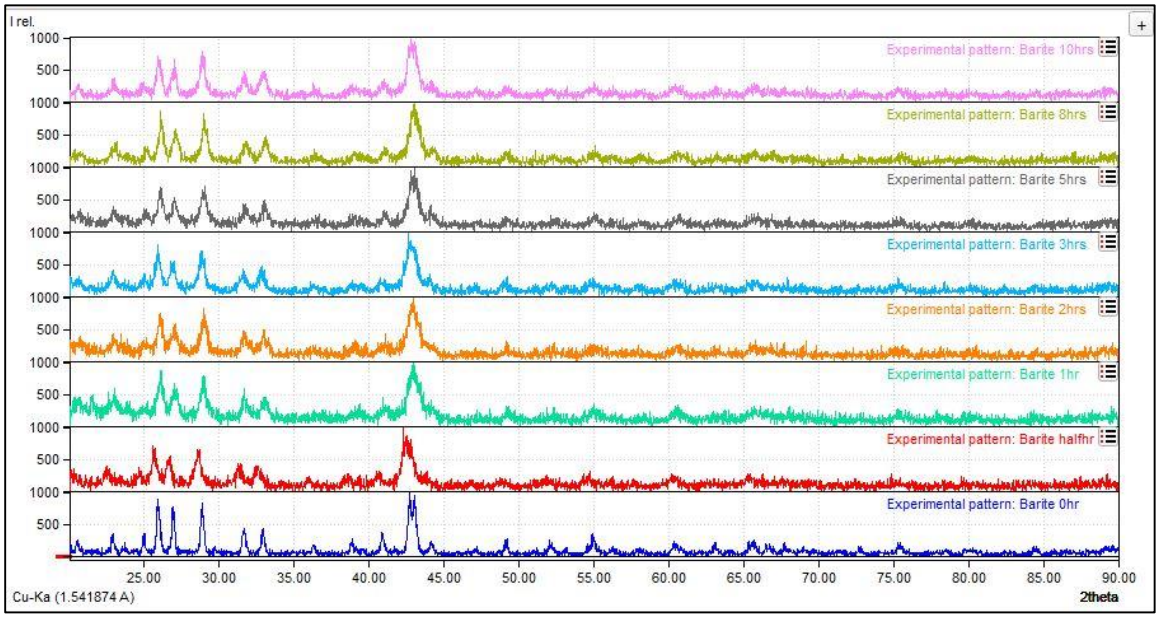

Figure 4.1: XRD Patterns of Barite Before and After Ball Milling

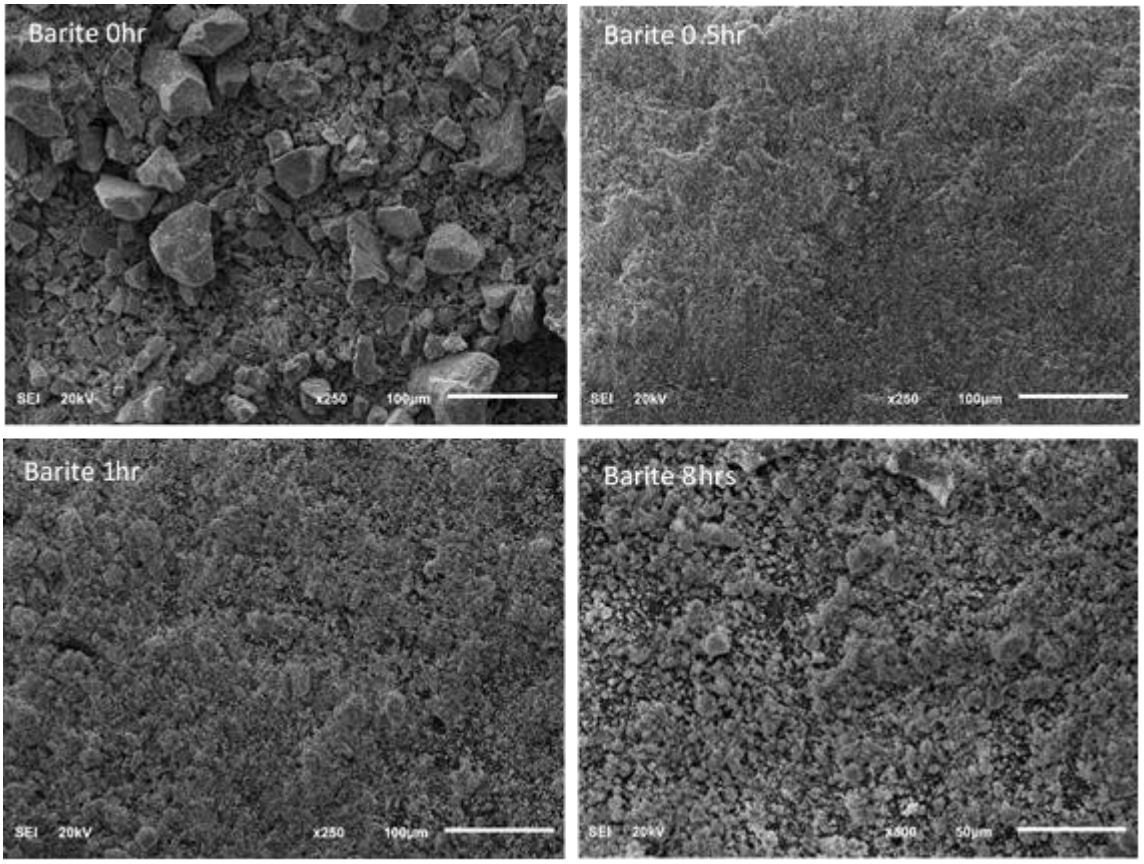

Figure 4.2: SEM Micrographs of Barite Before and After Ball Milling

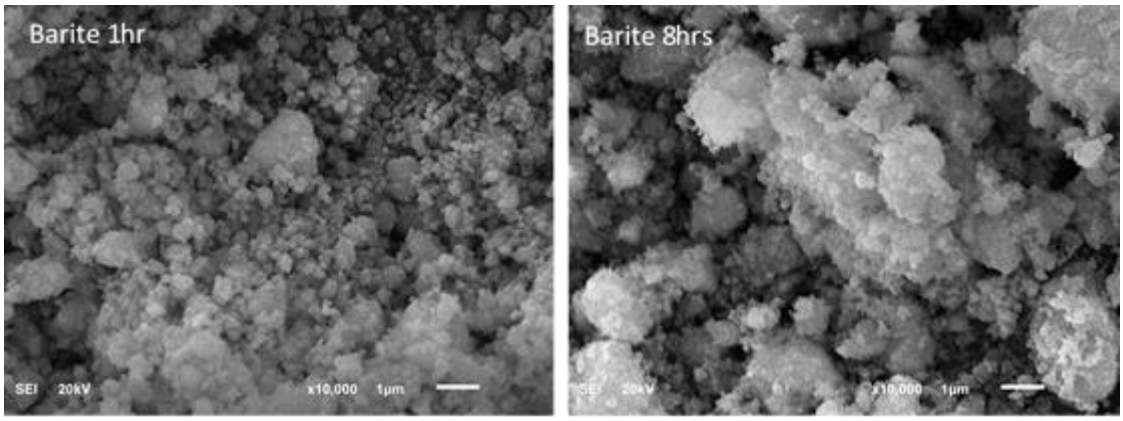

Figure 4.3: SEM Micrographs of Barite After Ball Milling for 1 and 8 Hours

Table 4.1: Barite Particle Size Before and After Ball Milling

Barite Sample	Peaks		
	Size (μm)	Volume %	Width
As Received, 0 hr	34.86	100	51.69
0.5 hr	175.3	28.1	228.3
	11.08	71.9	22.76
1 hr	18.85	100	95.81
3 hrs	63.08	61	124.6
	8.09	39	11.92
5 hrs	32.4	100	113.5

4.2 The Effect of Barite Particle Size on Fluid Stability

Stability is one of the crucial concerns to look at before introducing any fluid into the wellbore. As mentioned earlier, barite sag tendency is one of main drawbacks of using conventional barite-weighted fluids as completion fluids. Therefore, reducing barite particle size has been suggested as a solution to this phenomenon. The effect of barite particle size reduction on completion fluid stability was investigated using zeta potential and static sag test.

4.2.1 Zeta Potential Measurement

Zeta potential was measured over a wide range of pH to investigate the effect of barite particle size on fluid stability. Several samples of water were prepared with different pH, alkaline, neutral and acidic. Caustic soda and HCl were used as pH control agents. Different barite samples with different particle size were added to water samples with a concentration of 0.5 wt.%. Then these samples were placed in a multi-wrist shaker at room temperature for 20 to 24 hours to ensure that the charges equilibrium took place.

Afterwards, a sample from each solution was taken using a syringe with 5 µm filter. Zeta potential was measured using *ZetaPALS* zeta potential analyzer. An average value of zeta potential was calculated from 5 measurements, 20 runs for each reading.

As a result, it was found that for all barite samples: raw barite, 106-75 µm, and 75-40 µm, there was almost no enhancement in fluids stability over the whole range of pH, they were all lying with in the unstable range, ±30 mv, which indicates solids settlement. While barite sample of less than 40 µm was found to be stable at pH greater than 12. In contrast, micronized barite, around 1.5 µm, showed a moderate stability at pH values greater than 8 which includes the optimum range of pH at which conventional drilling fluids should be, i.e. 9-11. (**Figure 4.4**)

Although, zeta potential measurements were not conducted at the real field conditions, in terms of fluid composition, solids percent, temperature and pressure, these results gave an indication that reducing barite particle size to a few microns would improve fluid stability and minimize the risk of barite sag.

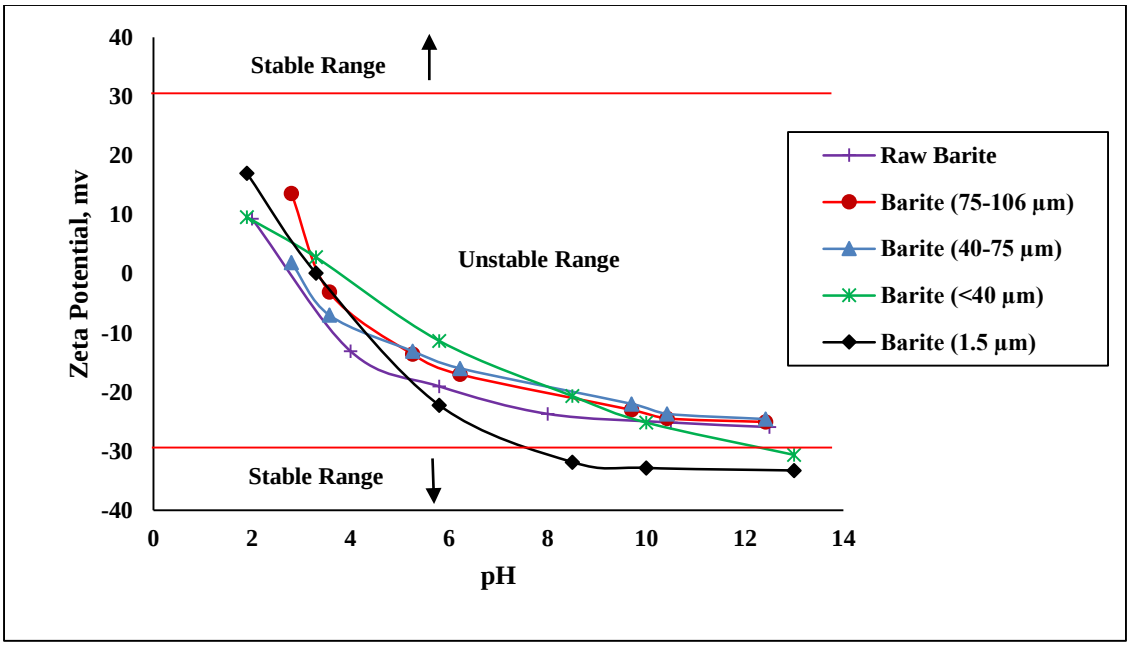

Figure 4.4: The Effect of Barite Particle Size on Zeta Potential Measurement

4.2.2 Sag Test

Unlike zeta potential, Sag test, to some extent, is the simulation of the real case. It was mainly performed to measure barite sag tendency to ensure completion fluids stability under HPHT conditions. First, different completion fluids were prepared using different barite particle size. Fluids were agitated using drilling fluid mixer. A Teflon vessel was used to contain the fluid and an aging cell was used to keep the fluid pressurized. A pressure of 500 psi was applied using nitrogen to prevent fluid from evaporation and the aging cell was put vertically in the oven for 24 hours at different temperatures. The test was conducted at static conditions. After 24 hours, the cell was taken out of the oven and left to cool down and the pressure was then released. Afterwards, two fluid samples were taken from the top and the bottom of the vessel. The densities of the two samples were measured and the difference in densities could be a qualitative tool to indicate fluid stability under that conditions.

4.2.2.1 Temperature Effect

First, the effect of temperature on barite sag was investigated at different temperatures, starting from room temperature up to 312 °F. Raw barite was used as weighting material. **Table 4.2** & **4.3** showed completion fluid formulation that was used in this test and its properties. At room temperature, there was almost no difference in densities and the sag factor was equal to 0.504 and as the temperature rose up, the difference in densities between the top and the bottom increased and sag performance was within the acceptable range (sag factor between 0.5 and 0.53) up to around 212 °F with a sag factor of 0.523. Beyond that temperature, the sag performance deteriorated and this could be attributed to Xanthan polymer degradation over temperature. **Figure 4.5** & **4.6** show the effect of temperature on completion fluid stability.

Furthermore, at a temperature of 312 °F, a complete degradation to the completion fluid was observed and completion fluid lost its viscosity and total solids settlement took place (**Figure 4.7**).

4.2.2.2 The Effect of Barite Particle Size

Different samples of completion fluid, using barite with different particle size as a weighting material, were prepared. The effect of barite particle size on sag performance was investigated. Sag test was conducted at a temperature of 230 °F and a pressure of 500 psi. The test was performed at vertical static conditions. **Figure 4.8** & **4.9** compared the difference between top and bottom densities, and sag factor for these different samples. These figures showed that there was no significant enhancement on fluid stability and all the samples had a sag factor out of the acceptable range, 0.5-0.53, therefore a bad sag

performance is highly anticipated at that conditions. Moreover, these results endorse the results of zeta potential measurement that reducing barite particle size to a few microns has impact on completion fluid stability however, this effect is not significant for a long time under the downhole conditions of high pressure and high temperature therefore, some dispersant for weighting material should be introduced to enhance completion fluid stability.

Table 4.2: Completion Fluid Formulation

Name	Description	Field Units/ Lab Units	Mixing Time, min	Function
Water	0.7	bbl/g	-	Base
Defoamer			-	Anti-Foam Agent
Soda ash	0.5	lb/g	10	Maintain Calcium Level
KOH	0.5	lb/g	10	pH Adjustment
Bentonite	4	lb/g	10	Viscosifier
XC-polymer	1-1.5	lb/g	20	Viscosifier
KCl	20	lb/g	10	Clay Stabilizer
CaCO3	5	lb/g	10	Bridging Agent
Barite	350	lb/g	10	Weighting Material
Sodium sulfite	0.25	lb/g	10	Oxygen Scavenger

Table 4.3: Completion Fluid Properties

Property	Unit	Value
Density	Pcf	115
Plastic Viscosity	cP	30
Yield Point	Lb/100 ft^2	35
Gel Strength, 10 sec	Lb/100 ft^2	20
Gel Strength, 10 min	Lb/100 ft^2	35
Gel Strength, 30 min	Lb/100 ft^2	45
pH	-	10

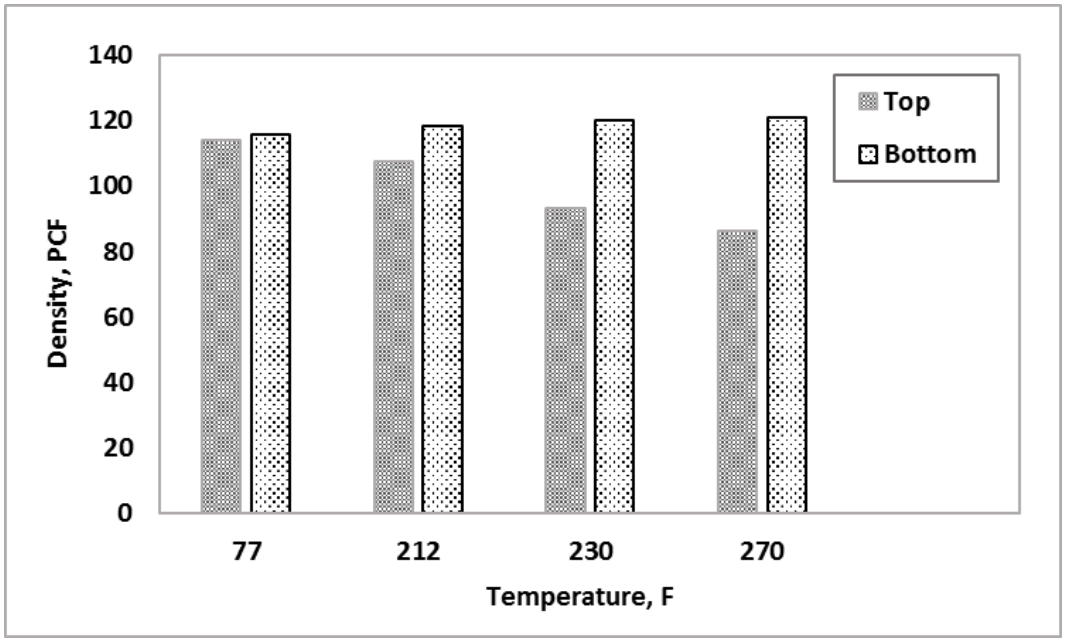

Figure 4.5: Temperature Effect on Densities Difference for Raw Barite After 24 Hours

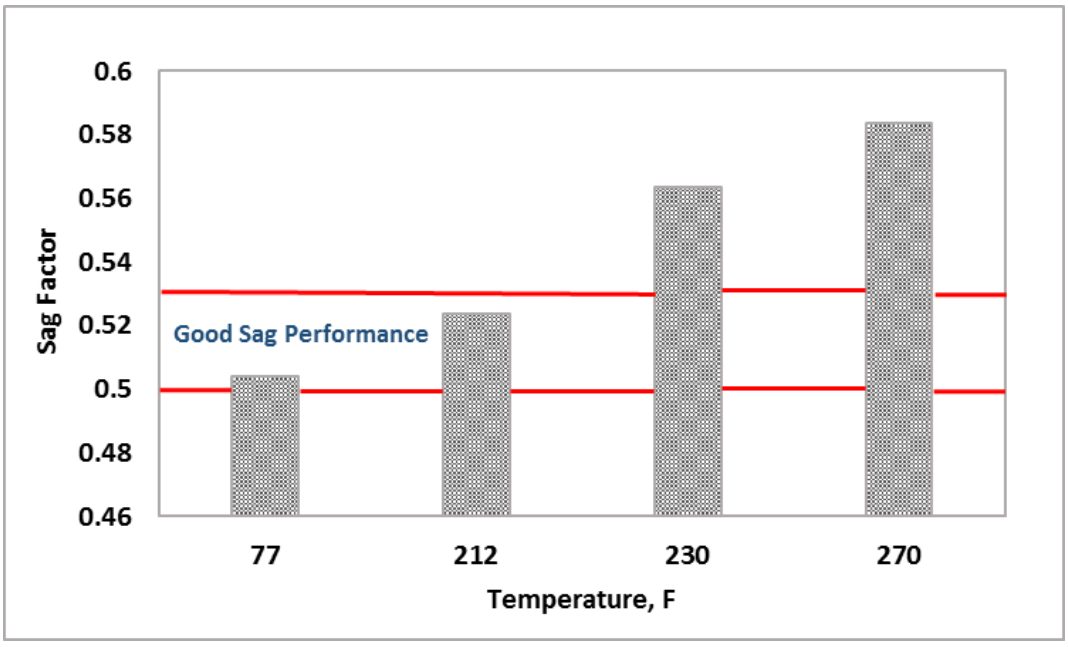

Figure 4.6: Temperature Effect on Sag Factor for Raw Barite After 24 Hours

Figure 4.7: A Sample of Completion Fluid After 24 Hours at 312 °F

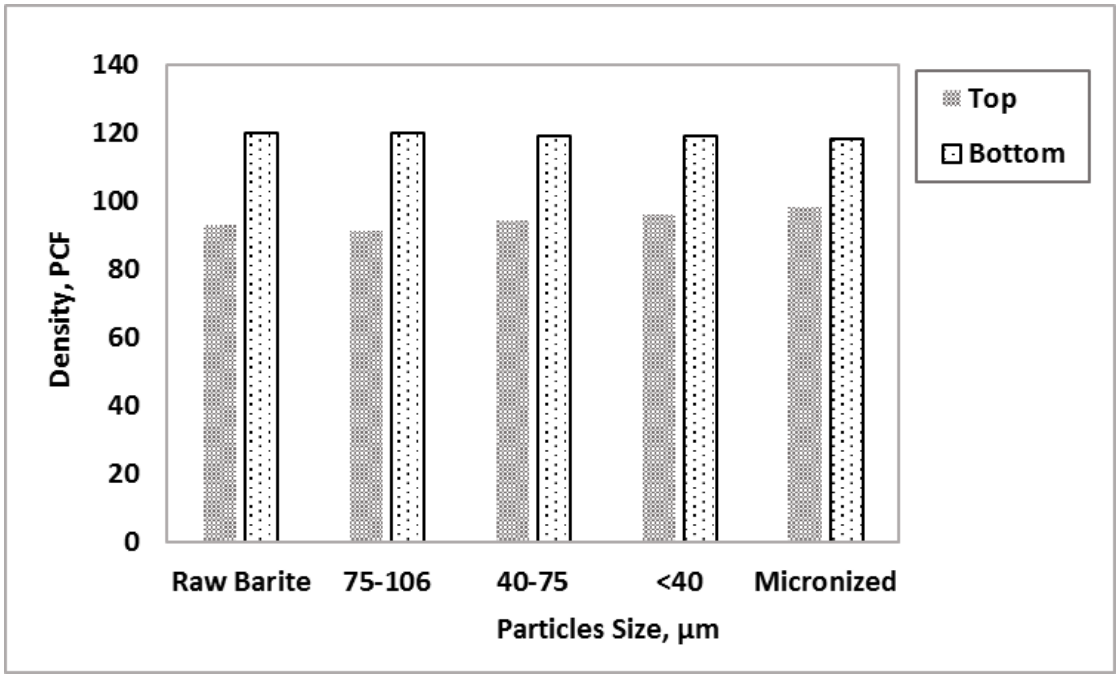

Figure 4.8: The Effect of Barite Particle Size on Densities Difference at 230 °F After 24 Hours

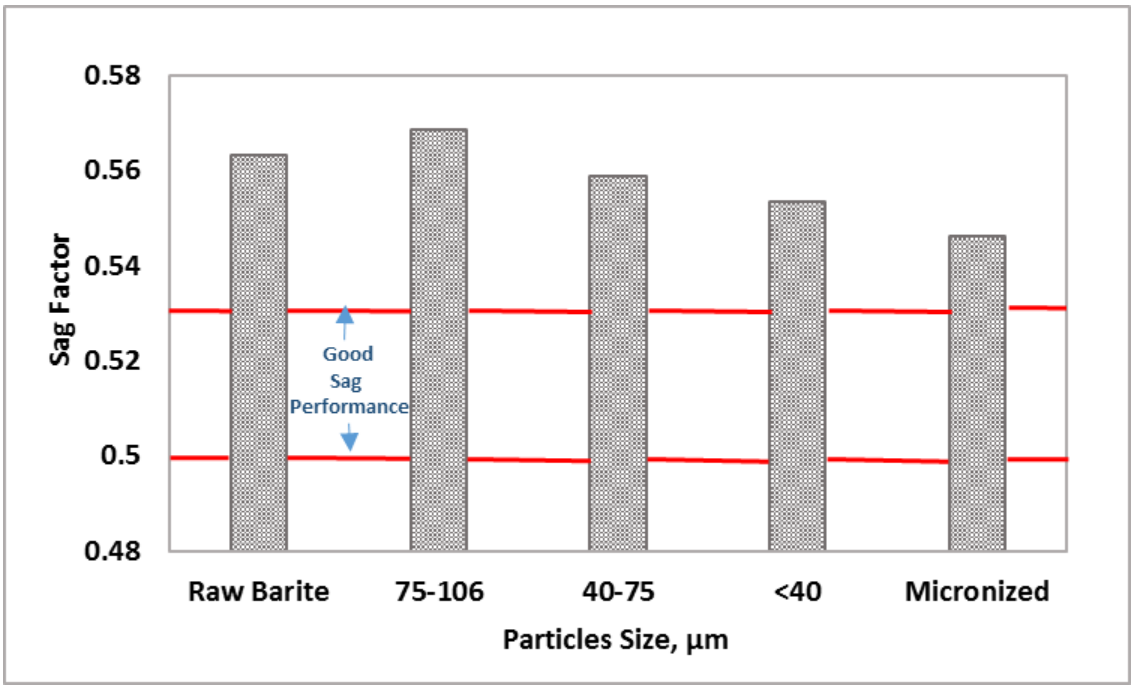

Figure 4.9: The Effect of Barite Particle Size on Sag Factor at 230 °F After 24 Hours

4.3 The Effect of Barite Particle Size on Rheology

Several samples of completion fluids were prepared using barite of different particle size as a weighting material. All samples have a density of 115 PCF. First, rheology was measured at 120 °F using *FAN 35A* rheometer. When fan readings (shear stress) were plotted against shear rate for all the samples, it was found that reducing barite particle size slightly shifted the curve up which indicates a thickening behavior and that could be attributed to the increase in surface area that consequently increase the friction between barite particles (**Figure 4.10**). **Figure 4.11** compares gel strength values after 10 seconds, 10 minutes, and 30 minutes for all fluid samples that were also measured at 120 °F. This figure also confirms the thickening behavior. This phenomenon can also confirm the results of zeta potential measurement and sag test that reducing barite particles size helps in improving fluid stability.

On the other hand, rheology was measured at 250 °F and 300 psi using *GRACE m5600* rheometer. Fan readings (shear stress) were plotted against shear rate for all fluid samples on **Figure 4.12**. As a result of high temperature, plastic viscosity decreased for all the samples and the discrepancy between the rheology curves of different barite particle size widened. Therefore, **Figure 4.10, 4.12** and **4.13** clarify that as the temperature rises up the effect of barite particle size on rheology becomes more noticeable. **Table 4.4** and **4.5** summarize the rheological properties of all completion fluid samples at 120 °F and 250 °F.

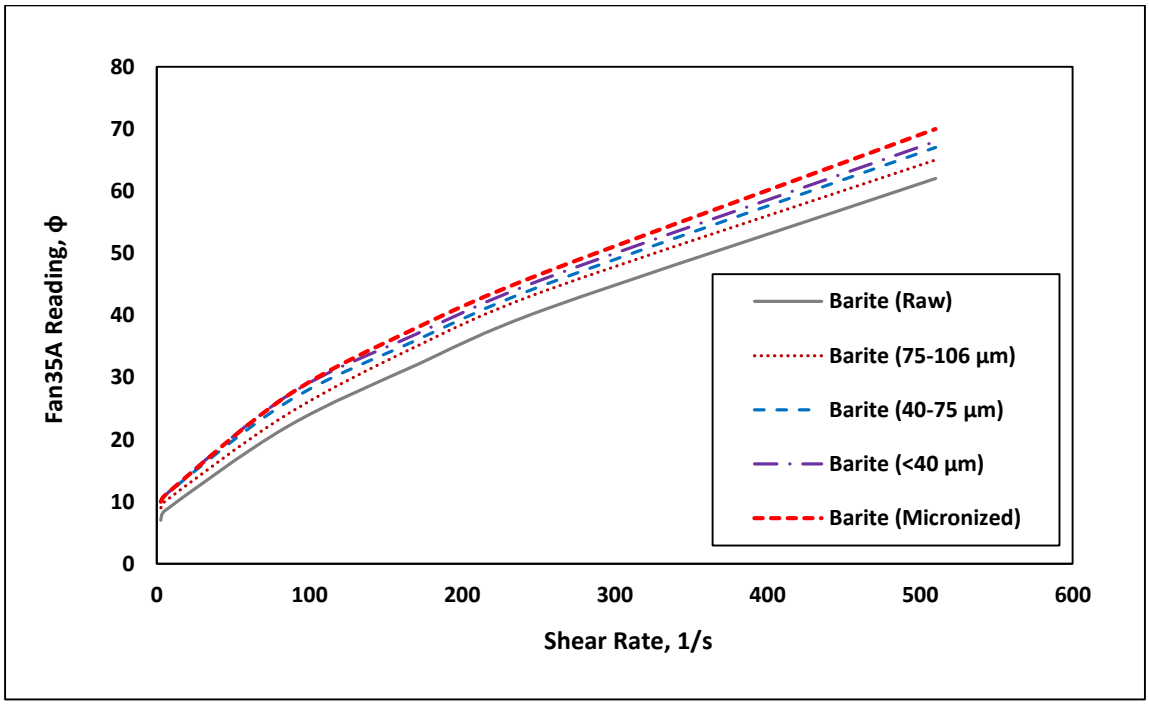

Figure 4.10: The Effect of Barite Particle Size on Rheology at 120 °F

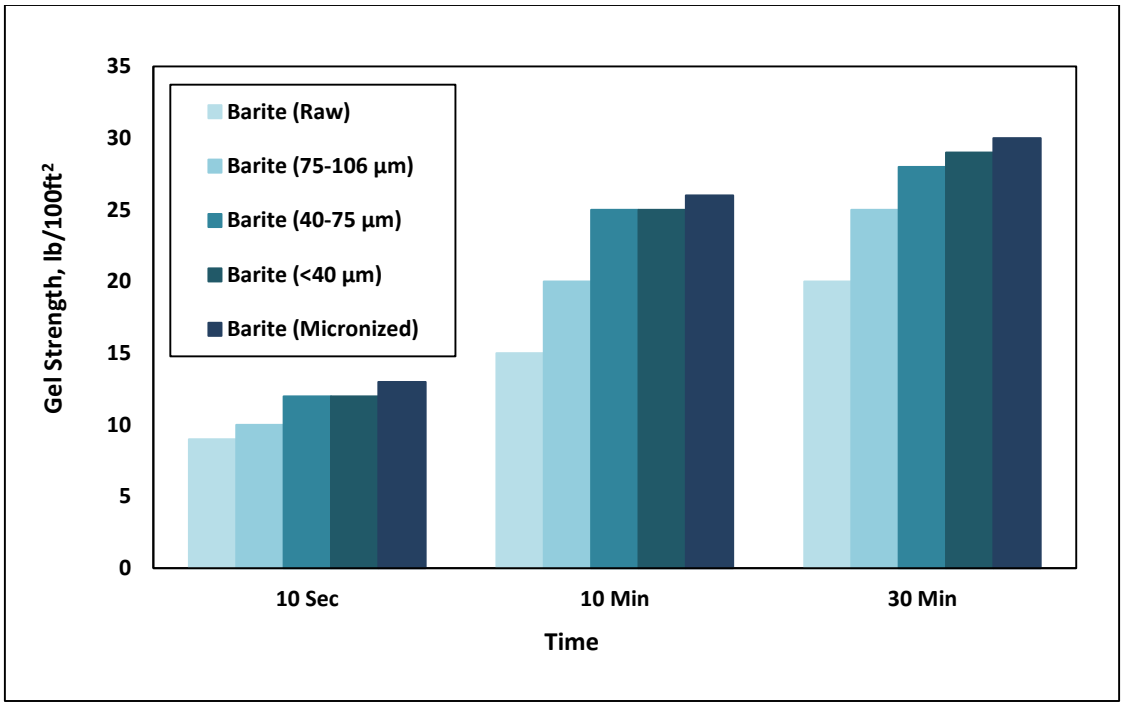

Figure 4.11: The Effect of Barite Particle Size on Gel Strength at 120 °F

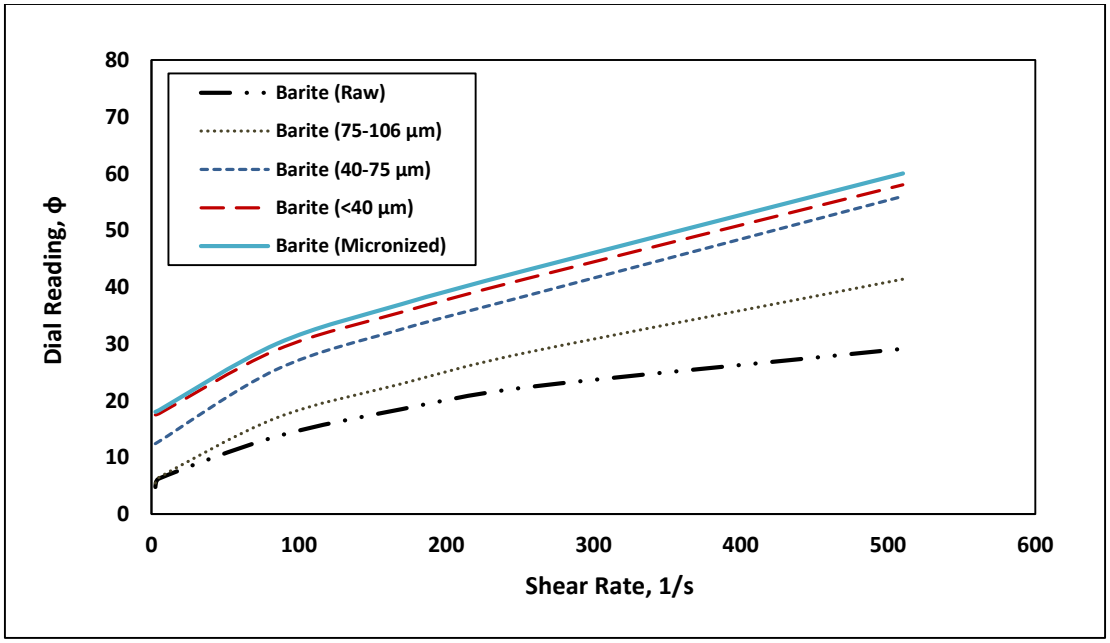

Figure 4.12: The Effect of Barite Particle Size on Rheology at 250 °F and 300 psi

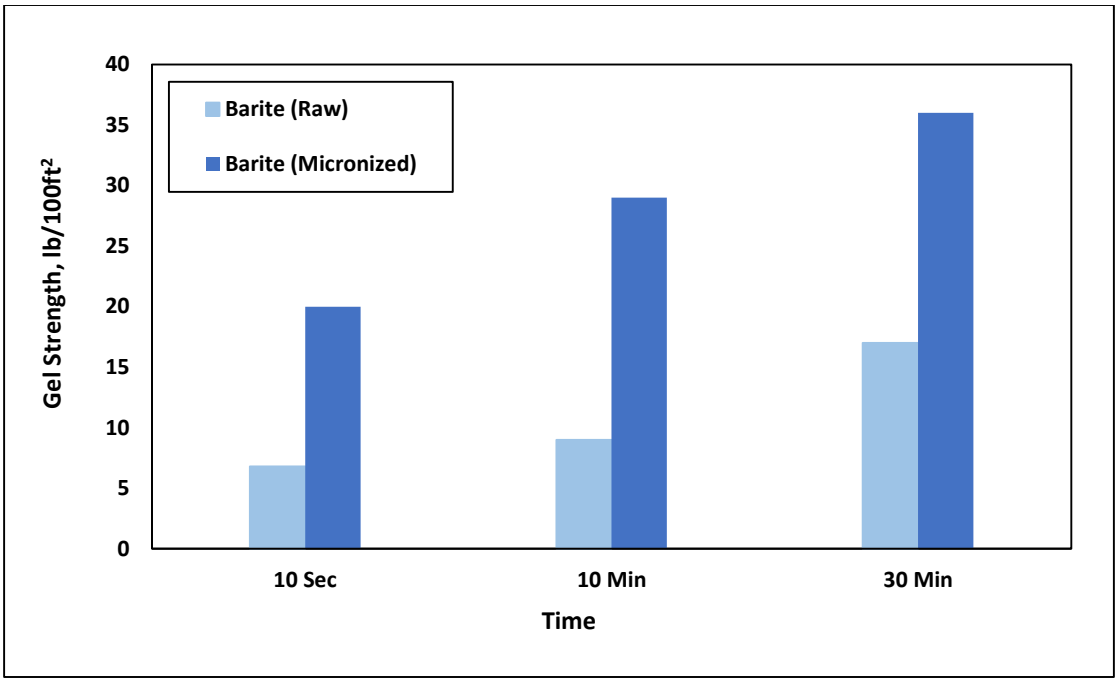

Figure 4.13: The Effect of Barite Particle Size on Gel Strength at 250 °F

Table 4.4: Completion Fluid Rheology at 120 °F

Property	Raw Barite	Barite 75-106 µm	Barite 40-75 µm	Barite <40 µm	Micronized Barite
Density, PCF	115	115	115	115	115
Plastic Viscosity, cP	21	21	22	22	23
Yield Point, Lb/100 ft²	20	23	23	24	24
Gel Strength (10 sec), Lb/100 ft²	9	10	12	12	13
Gel Strength (10 min), Lb/100 ft²	15	20	25	25	26
Gel Strength (30 min), Lb/100 ft²	20	25	28	29	30
pH	10	10	10	10	10

Table 4.5: Completion Fluid Rheology at 250 °F

Property	Raw Barite	Barite 75-106 µm	Barite 40-75 µm	Barite <40 µm	Micronized Barite
Density, PCF	115	115	115	115	116
Plastic Viscosity, cP	6.75	12.9	17.5	16.5	17
Yield Point, Lb/100 ft²	15.6	15.6	21	25	26
Gel Strength (10 sec), Lb/100 ft²	6.8	7	13	18	20
Gel Strength (10 min), Lb/100 ft²	9	12	24	27	29
Gel Strength (30 min), Lb/100 ft²	17	19	31	35	36
pH	10	10	10	10	10

4.4 The Effect of Particle Size on Barite Removal

4.4.1 Solubility Test

Solubility test was performed to investigate the effect of particle size on barite removal. A new formulation that has been introduced by Ba Geri et. al. (2016) was used. This formulation is mainly based on DTPA chelating agent at high pH in addition to potassium carbonate as a catalyst. All the experiments were conducted with a ratio of 1 gram to 20 ml at a temperature of 312 °F at dynamic condition.

Figure 4.14 & **4.15** show the results of solubility test of barite with different particle size in DTPA with potassium carbonate. These figures show a slight increase in barite solubility as the particle size decreases and this is due to the fact that reducing barite particle size increases the surface area therefore, enhances the chemical reaction. An enhancement of around 11 g/L in solubility was observed between the ball milled sample of barite to the largest sample that was obtained using sieve analysis, 75-106 µm.

Another sample of barite was tested to ensure the effectiveness of particle size reduction on barite removal. The new sample has an average particle size of 1.5 microns that was measured using *Turbtrac* particle size analyzer. **Table 4.6** and **4.7** show the chemical composition of both samples of barite that was determined using XRF technique. **Figure 4.16** compared the solubility of the two samples in percentage and gram per liter. It shows a small discrepancy in barite solubility, around 1 g/L, that could be attributed to the difference in chemical composition of both samples of barite. Thus, it confirms the effectiveness of reducing barite particle size.

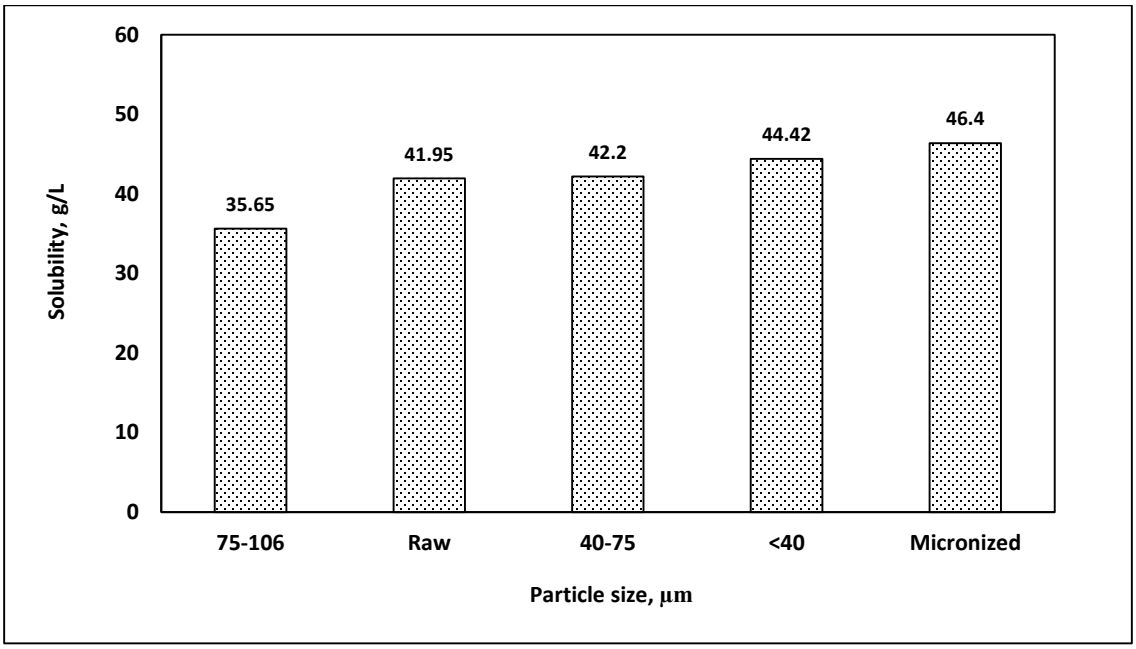

Figure 4.14: The Effect of Particle Size on Barite Solubility (g/L) in DTPA with K_2CO_3

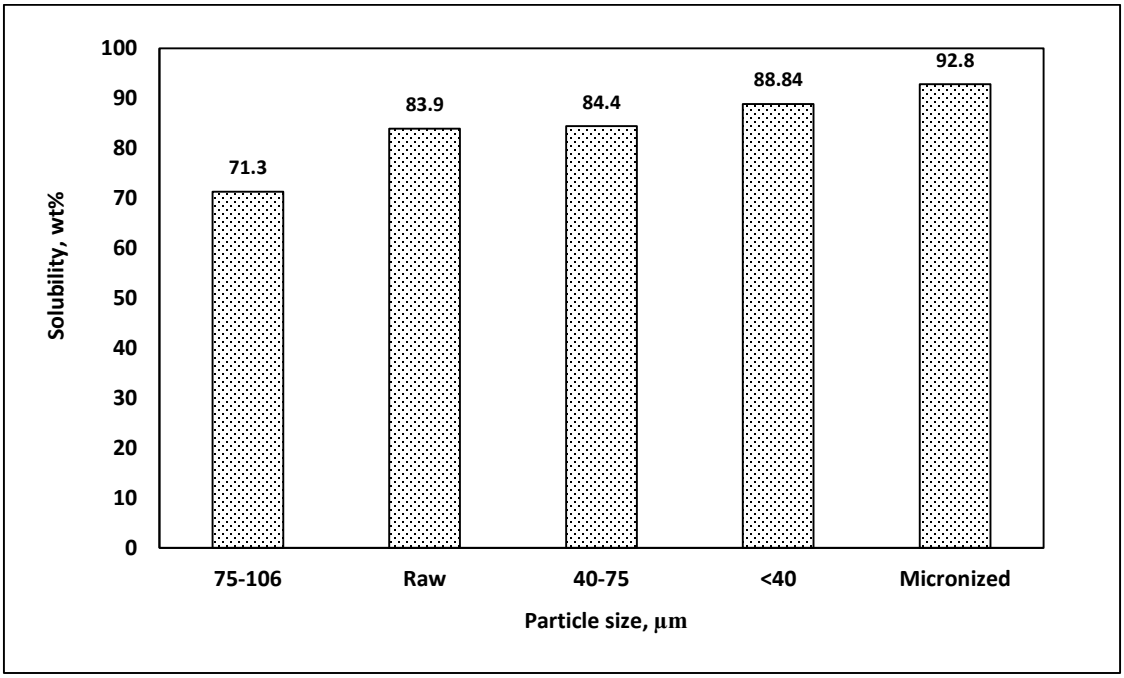

Figure 4.15: The Effect of Particle Size on Barite Solubility (%) in DTPA with K_2CO_3

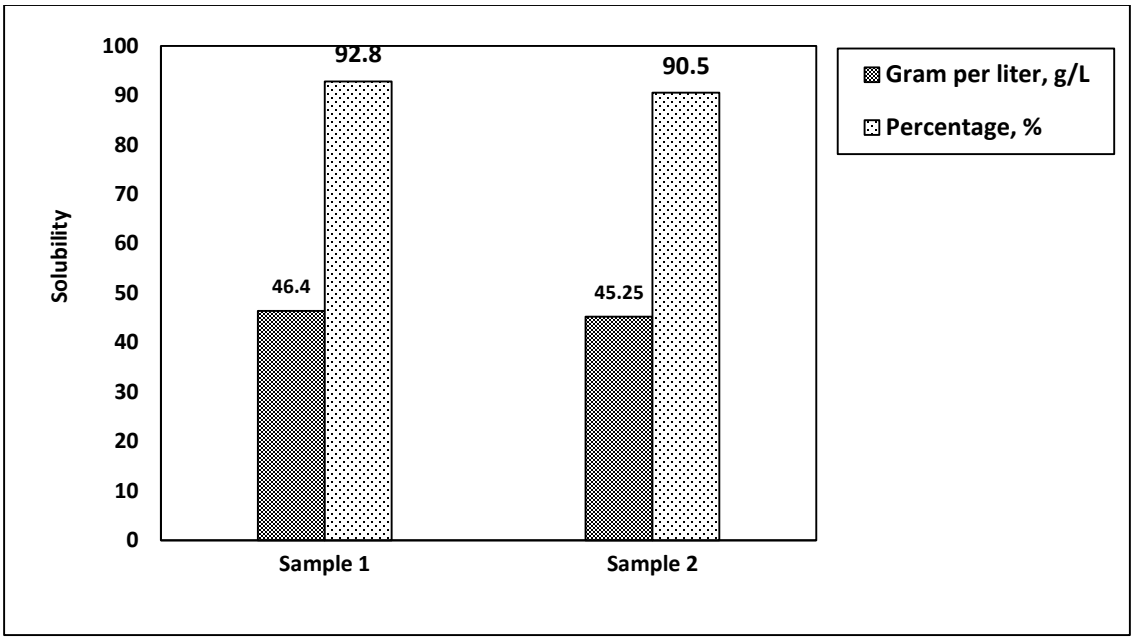

Figure 4.16: Barite Solubility in DTPA with K_2CO_3 for Two Different Barite Samples

Table 4.6: Chemical Composition of Barite Samples (Oxides)

Element	Sample 1		Sample 2	
	wt.%	mol%	wt.%	mol%
SiO2	3.5793	7.0605	1.8825	3.7033
SO3	25.1894	37.2893	28.9102	42.6802
K2O	0.5714	0.7189	-	-
CaO	0.1328	0.2806	0.0862	0.1816
Fe2O3	1.3336	0.9898	0.8381	0.6203
CuO	0.037	0.0551	0.0359	0.0534
SrO	0.4619	0.5283	0.7157	0.8164
MoO	0.0121	0.0128	0.0059	0.0062
BaO	68.6162	53.0401	67.0289	51.6711
Ta2O5	0.0411	0.011	-	-
PbO	0.0254	0.0135	0.4291	0.2272
ZnO	-	-	0.0192	0.0278
Re2O7	-	-	0.0177	0.0043
Au2O3	-	-	0.0308	0.0082

Table 4.7: Chemical Composition of Barite Samples (Elements)

Element	Sample 1		Sample 2	
	wt.%	mol%	wt.%	mol%
Si	1.9916	6.3483	1.1527	3.6047
S	12.6341	35.275	15.2679	41.8223
K	0.6331	1.4498	-	-
Ca	0.1109	0.2478	0.0789	0.1729
Fe	1.3338	2.1383	0.8188	1.2879
Ni	0.0157	0.024	-	-
Cu	0.0354	0.0499	0.0392	0.0542
Sr	0.5518	0.5638	0.8483	0.8504
Mo	0.017	0.0159	-	-
Ba	82.6171	53.8602	81.2085	51.9405
Ta	0.023	0.0114	-	-
Pb	0.0366	0.0158	0.5652	0.2396
Zn	-	-	0.0205	0.0275

4.4.2 HPHT Filtration Test

Filtration test was conducted to evaluate the effect of reducing barite particle size on fluid filtration performance and barite removal which consequently affect the resulted formation damage. Two samples of completion fluid were prepared using barite of two different sizes as weighting material, raw barite and micronized barite of around 5 µm. Both samples have the same fluid formulation (**Table 4.2**). Different proportions of calcium carbonate (medium and fine with a ratio of 3.5 to 1.5 grams, respectively) were combined to mitigate solid particles invasion with micronized barite. Filtration test was performed at static conditions at 250 °F and 300 psi.

Figure 4.17 shows the filtration performance of both fluid samples of micronized barite. The old formulation of completion fluid using calcium carbonate with medium particle size as a bridging agent, while the new formulation was designed by combining two proportions of calcium carbonate (medium and fine with a ratio of 3.5 to 1.5 grams, respectively). This figure

shows that reducing barite particle size increases the amount of fluid filtrate into the formation and therefore increases the possibility of formation damage due to the poor bridging that resulted from reducing the particle size. Moreover, the old formulation failed to build a proper filter cake, thin and impermeable, while combining different proportions of calcium carbonate which works as bridging agent helped in minimizing the fluid filtrate to its minimum and the difference in filtrate volume between raw and micronized barite was reduced to only around 1 cc and good filter cake was built (**Figure 4.18**).

In removal part, DTPA (20 wt.%) of high pH was used to remove filter cake. 6 wt.% of potassium carbonate was added to DTPA as a catalyst and 7 wt.% of enzyme was added to break the polymer. Removal part was performed at the same condition of temperature and pressure and it was run for 24 hours. As a result of removal test, reducing barite particle size to a few microns enhanced the removal efficiency by 5%, from 84.3% for raw barite to 89.23% for micronized barite which confirmed the results of solubility test. Nevertheless, there is a difference in barite removal percentage between solubility test and HPHT filtration test and this discrepancy is due to the presence of other additives in completion fluid and the difference in experimental conditions for each test. **Table 4.8** summarizes the results of HPHT filtration test and **Figure 4.19** shows the resulted filter cake before and after removal.

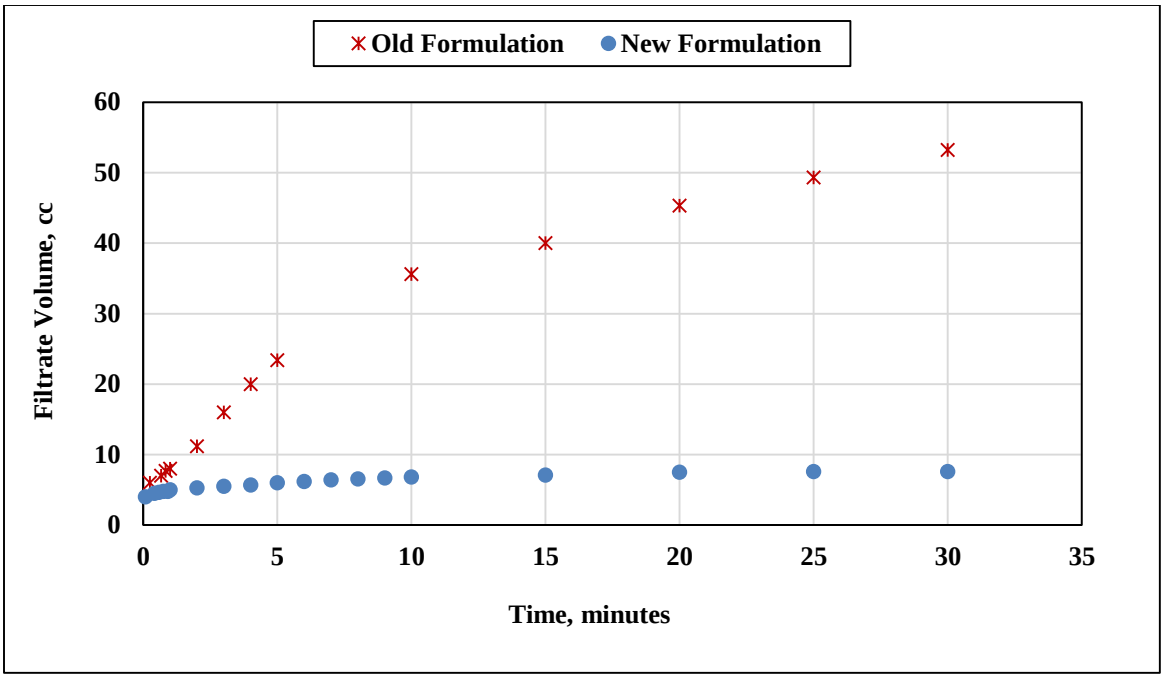

Figure 4.17: The Effect of Adding Different Proportions of Bridging Agent on Filtration Performance of Micronized Barite

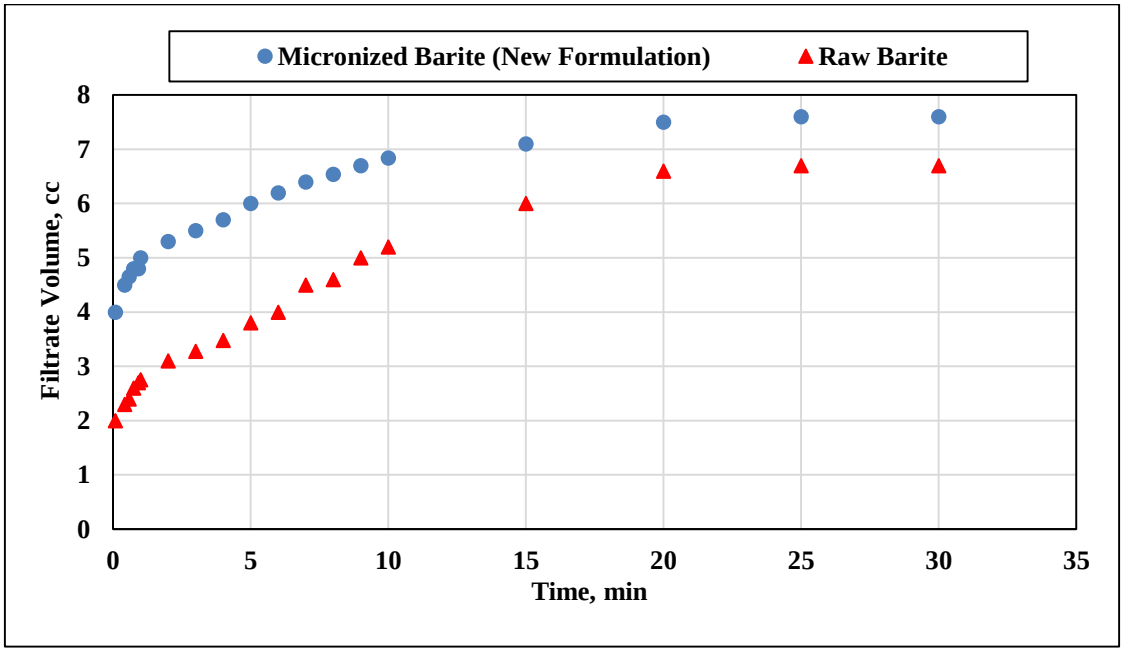

Figure 4.18: Filtration Performance of Raw and Micronized Barite Samples

Table 4.8: HPHT Filtration Test Summary

Parameter	Raw Barite	Micronized Barite
Filtrate, cm^3/30min	6.7	7.6
Filter-cake Weight, g	20.33	21.35
Removed filter cake, g	17.14	19.05
Removal Efficiency, wt.%	84.3	89.23

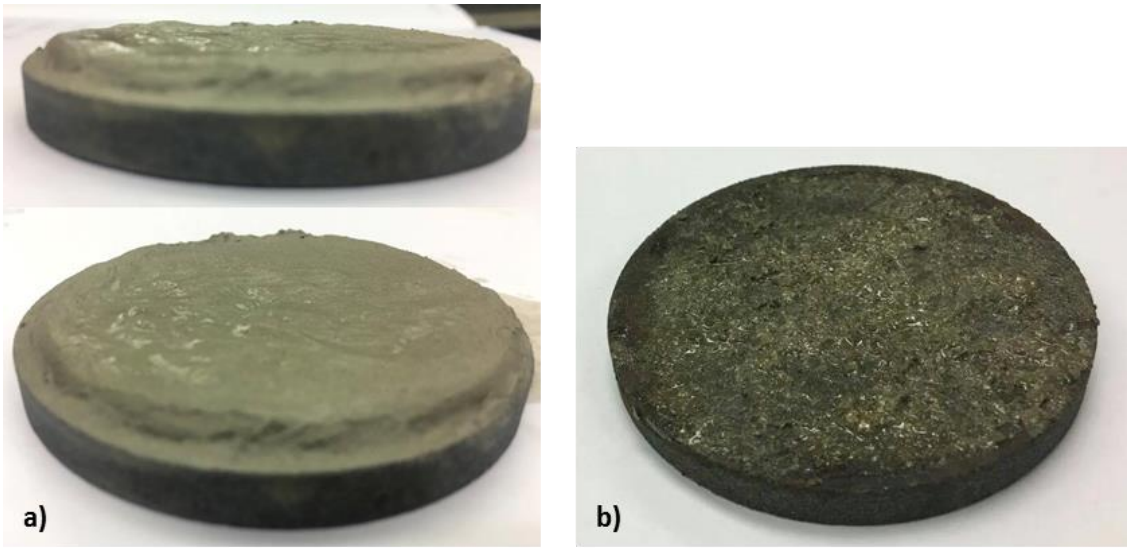

Figure 4.19: Filter-cake Before (a) and After Removal (b)

CHAPTER V

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

In this study, different samples of barite with different particle size ranges were used to investigate the effect of barite particle size on completion fluids performance at high pressure high temperature conditions (HPHT). These samples were obtained using sieve analysis and ball milling.

Ball milling experiments showed small particles agglomeration after milling for a long time and that was confirmed by SEM micrographs and particle size analysis. Milling time of half an hour was used to produce the micronized barite sample. And another sample of micronized barite with an average particle size of 1.5 micron was used also to evaluate micronized barite as a completion fluid weighting material.

These barite samples were added to deionized water with a wide range of pH with a concentration of 5 wt.%. HCL and NaOH were used as pH control agents. Suspensions were left for 20-24 hours before measuring zeta potential. Zeta potential measurements showed that reducing barite particle size to a few microns improved the fluid stability and moderate stability was observed for pH range greater than 8 which includes the normal range of completion and drilling fluids. In contrast, all the other sizes of barite were lying in the unstable range (±30 mv) and no enhancement in fluid stability was observed.

Completion fluids were prepared with a density of 115 pcf and rheological properties were measured at 120 and 250 °F. Rheology measurements showed a thickening behavior as barite particle size decreased and this behavior was more noticeable at high temperatures.

Completion fluid formulation showed a good sag performance up to around 215 °F and as the temperature rises up sag performance deteriorates until 270 °F. Beyond that temperature a complete degradation to Xanthan polymer occurred and total solids settlement occurred. Sag test results also showed insignificant improvement in sag performance as the particle size was reduced.

Diethylene tri-amine penta-acetic acid (DTPA) with potassium carbonate was used to remove barite with a concentration of 20 wt.% and 6 wt.%, respectively. Barite was added to this formulation with a ratio of 1 gram to 20 ml. Solubility test results showed a good enhancement in barite removal as the particle size was reduced with a difference of around 11 g/L (20 wt.%) between the largest and the micronized size of barite.

HPHT Filtration test confirmed that combining different sizes of bridging agent (calcium carbonate) plays a great role in minimizing the volume of fluid filtrate into the formation and there for mitigate the risk of formation damage. It also confirmed the solubility test results and an enhancement of 5% in removal efficiency was recorded as barite particle size was reduced to few microns.

5.2 Recommendations

1. Study the effect of reducing barite particle size to Nano size on fluid stability.
2. Study the effect of adding ilmenite to barite as a weighting material on enhancing fluid stability.
3. Adding new Additives, polymer or other chemicals, that could help in barite dispersion to enhance completion fluids stability and mitigate barite sag tendency.

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APPENDIX

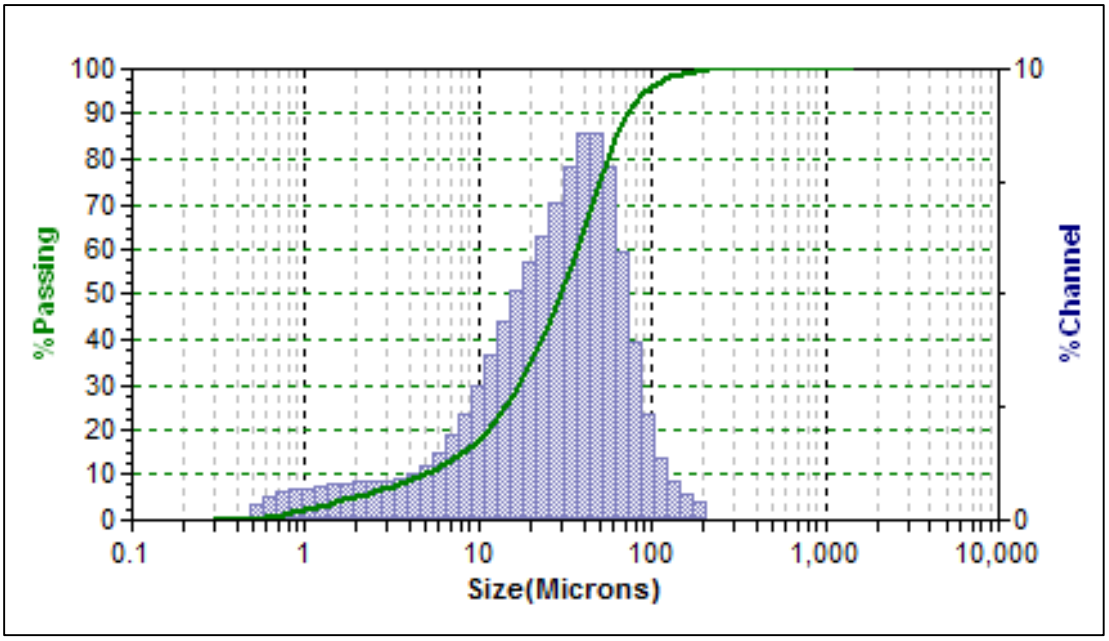

Figure A.1: Particle Size Distribution of Barite Sample (106-75 µm)

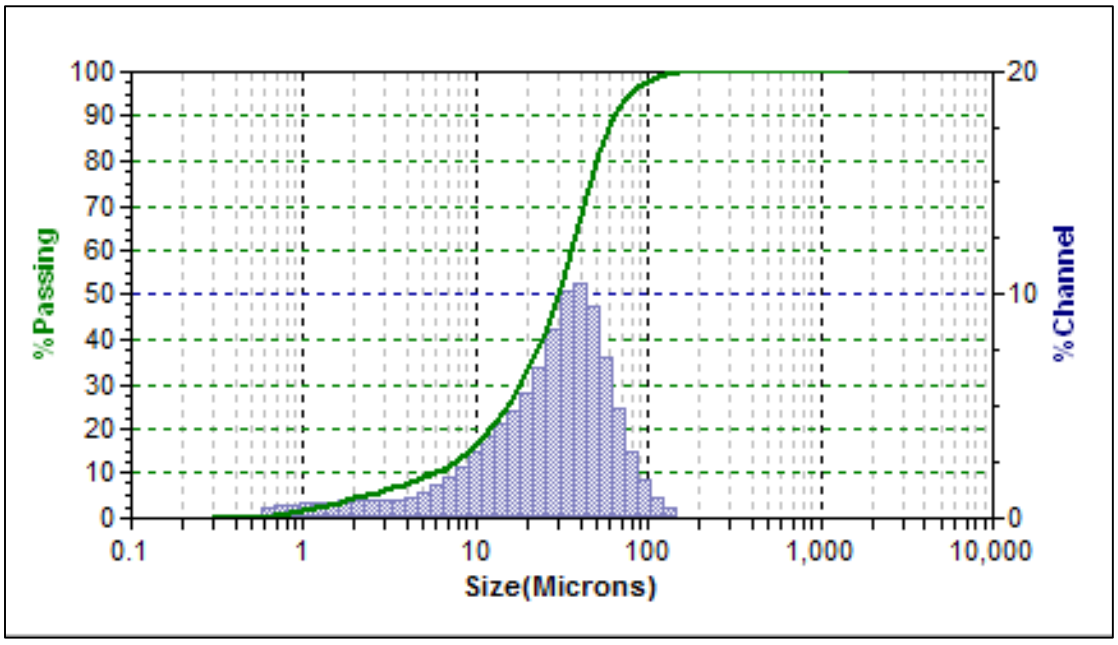

Figure A.2: Particle Size Distribution of Barite Sample (75-40 µm)

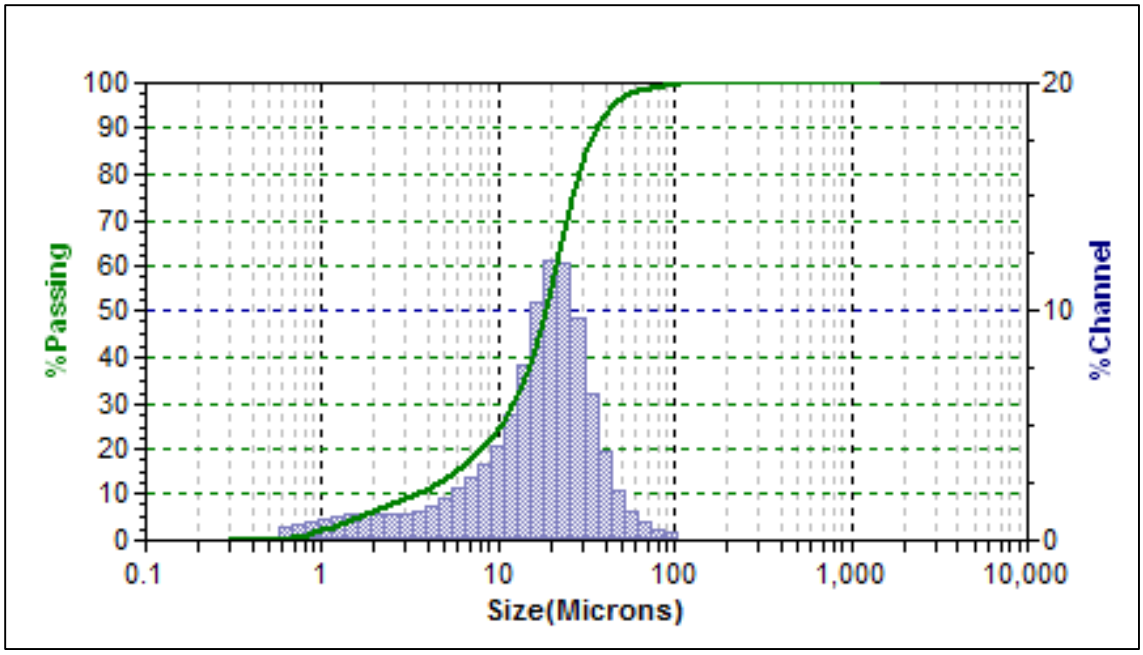

Figure A.3: Particle Size Distribution of Barite Sample (Less than 40 µm)

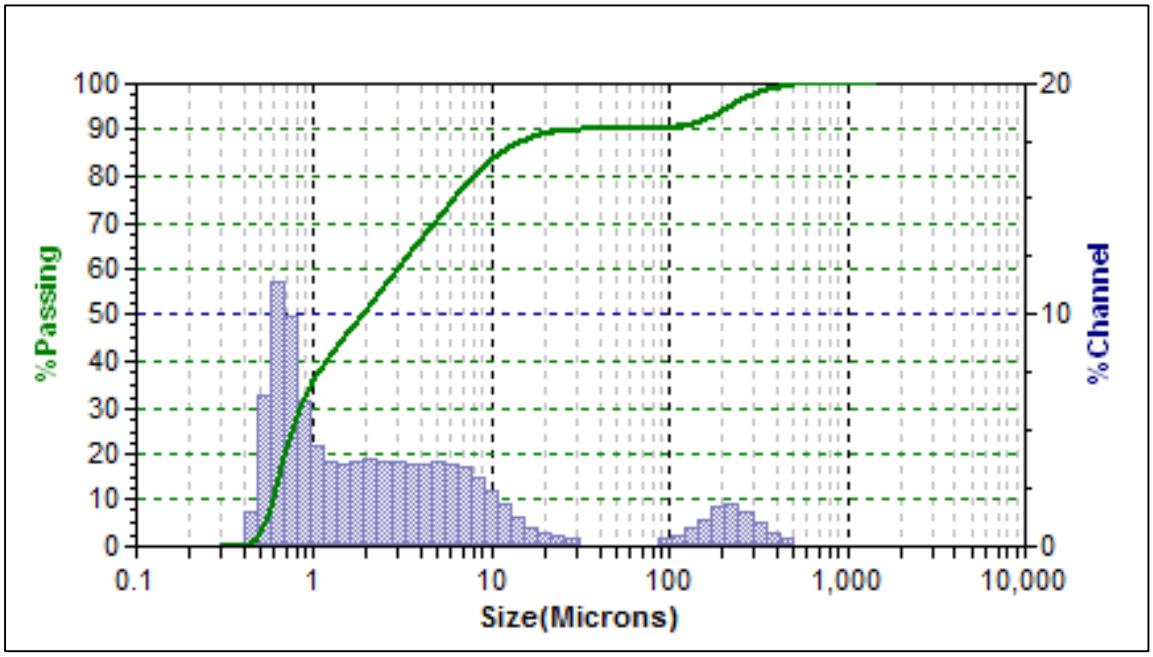

Figure A.4: Particle Size Distribution of Micronized Barite Sample

Table A.1: Zeta Potential Measurements of Raw Barite

pH	Zeta
2	9.31
4	-13.11
5.8	-19.06
8	-23.71
10.5	-25.17
12.5	-25.94

Table A.2: Zeta Potential Measurements of Barite (106-75 µm)

pH	Zeta
2.8	13.6
3.57	-3.1
5.26	-13.61
6.22	-17
9.7	-23
10.42	-24.44
12.42	-25.04

Table A.3: Zeta Potential Measurements of Barite (75-40 µm)

pH	Zeta
2.8	1.9
3.57	-7
5.26	-13.1
6.22	-16
9.7	-22.01
10.42	-23.7
12.42	-24.59

Table 5.4: Zeta Potential Measurements of Barite (<40 µm)

pH	Zeta
1.9	9.59
3.3	2.8
5.8	-11.35
8.5	-20.7
10	-25.18
13	-30.67

Table A.5: Zeta Potential Measurements of Micronized Barite (1.5 µm)

pH	Zeta
1.9	17.03
3.3	0.11
5.8	-22.22
8.5	-31.86
10	-32.85
13	-33.28

Table A.6: Filtrate Volume with Time for Micronized Barite at 250 °F (Old Formulation)

Time, min	Filtrate volume, cc
0.25	6
0.67	7
0.84	7.7
1	8
2	11.2
3	16
4	20
5	23.4
10	35.6
15	40
20	45.3
25	49.3
30	53.2

Table A.7: Filtrate Volume with Time for Micronized Barite at 250 °F (New Formulation)

Time, min	Filtrate volume, cc
0.08	4
0.42	4.5
0.58	4.65
0.75	4.8
0.92	4.8
1	5
2	5.3
3	5.5
4	5.7
5	6
6	6.2
7	6.4
8	6.54
9	6.7
10	6.84
15	7.1
20	7.5
25	7.6
30	7.6

Table A.8: Filtrate Volume with Time for Raw Barite at 250 °F

Time, min	Filtrate volume, cc
0.08	2
0.42	2.3
0.58	2.4
0.75	2.6
0.92	2.7
1	2.75
2	3.1
3	3.28
4	3.48
5	3.8
6	4
7	4.5
8	4.6
9	5
10	5.2
15	6
20	6.6
25	6.7
30	6.7

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